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U. S. DEPARTMENT OF AGRICULTURE,

BUREAU OF CHEMISTRY—BULLETIN No. 107 (Revised).

H. W. WILEY, Chief of Bureau.

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# OFFICIAL AND PROVISIONAL METHODS OF ANALYSIS,

ASSOCIATION OF OFFICIAL  
AGRICULTURAL CHEMISTS.

AS COMPILED BY THE COMMITTEE ON REVISION OF METHODS (J. K. HAYWOOD,  
CHAIRMAN, J. P. STREET, F. W. WOLL, J. H. PETTIT, L. M. TOLMAN,  
F. P. VEITCH, AND A. L. WINTON), NOVEMBER 1, 1906, AND  
PROVISIONALLY ADOPTED BY THE ASSOCIATION;  
FURTHER REVISED AND FINALLY  
ADOPTED, OCTOBER, 1907.

Edited by  
HARVEY W. WILEY,  
Secretary of the Association.



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## LETTER OF TRANSMITTAL FOR REVISED EDITION.

U. S. DEPARTMENT OF AGRICULTURE,  
BUREAU OF CHEMISTRY,  
*Washington, D. C., April 20, 1908.*

SIR: At the twenty-fourth annual convention of the Association of Official Agricultural Chemists the following resolution was passed in regard to the report of the committee on the revision of methods, submitted for approval as Bulletin 107 of the Bureau of Chemistry:

*Resolved*, That the report of the committee on revision, as submitted, be approved and the committee continued, with the authority to issue the final revision and to make such additional changes as they deem necessary, with the approval of the executive committee.

In accordance with these instructions, Bulletin 107, revised, herewith, is submitted for publication, additional changes as suggested by the association having been made with the approval of the executive committee, and all action in regard to methods taken at the meeting of 1907 having been incorporated.

Respectfully,

H. W. WILEY,  
*Secretary, Association of Official Agricultural  
Chemists; Chief, Bureau of Chemistry.*

HON. JAMES WILSON,  
*Secretary of Agriculture.*

## LETTER OF TRANSMITTAL FOR ORIGINAL EDITION.

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U. S. DEPARTMENT OF AGRICULTURE,

BUREAU OF CHEMISTRY,

*Washington, D. C., July 15, 1907.*

SIR: In view of the increasing importance, legally as well as scientifically, of the methods of the Association of Official Agricultural Chemists, in connection with the administration of the food and drugs act, June 30, 1906, these methods being also those used in the Bureau of Chemistry, it seemed of fundamental importance that they should be revised to date, no revision having been issued since 1899, with the exception of the record made in additional circulars and in Bulletin 65 on food analysis, of the additions to and changes in the methods.

The difficulties of making such a revision, when the methods, especially those relating to foods, are continually the subject of study and amendment, are apparent. Nevertheless it is believed that the present revision, including all authorized changes and additions, the rearrangement and consolidation of general and special methods, and the elimination of repetitions or contradictory material, will so add to the efficiency and usefulness of the methods as to warrant its publication, although many other changes might be suggested.

The revision is the work of the following committee, appointed by the president of the association, previous to the meeting of 1906, and provisionally adopted at that time: J. K. Haywood, chairman; J. P. Street, F. W. Woll, J. H. Pettit, L. M. Tolman, F. P. Veitch, and A. L. Winton. The preliminary work on the revision was done by members of the Bureau of Chemistry.

It is respectfully recommended that this manuscript be issued as Bulletin 107 of the Bureau of Chemistry, superseding Bulletins 46 and 65, the revision to be in this form submitted to the association for final approval at the meeting of 1907.

Respectfully,

H. W. WILEY,

*Secretary, Association of Official Agricultural  
Chemists; Chief, Bureau of Chemistry.*

HON. JAMES WILSON,

*Secretary of Agriculture.*

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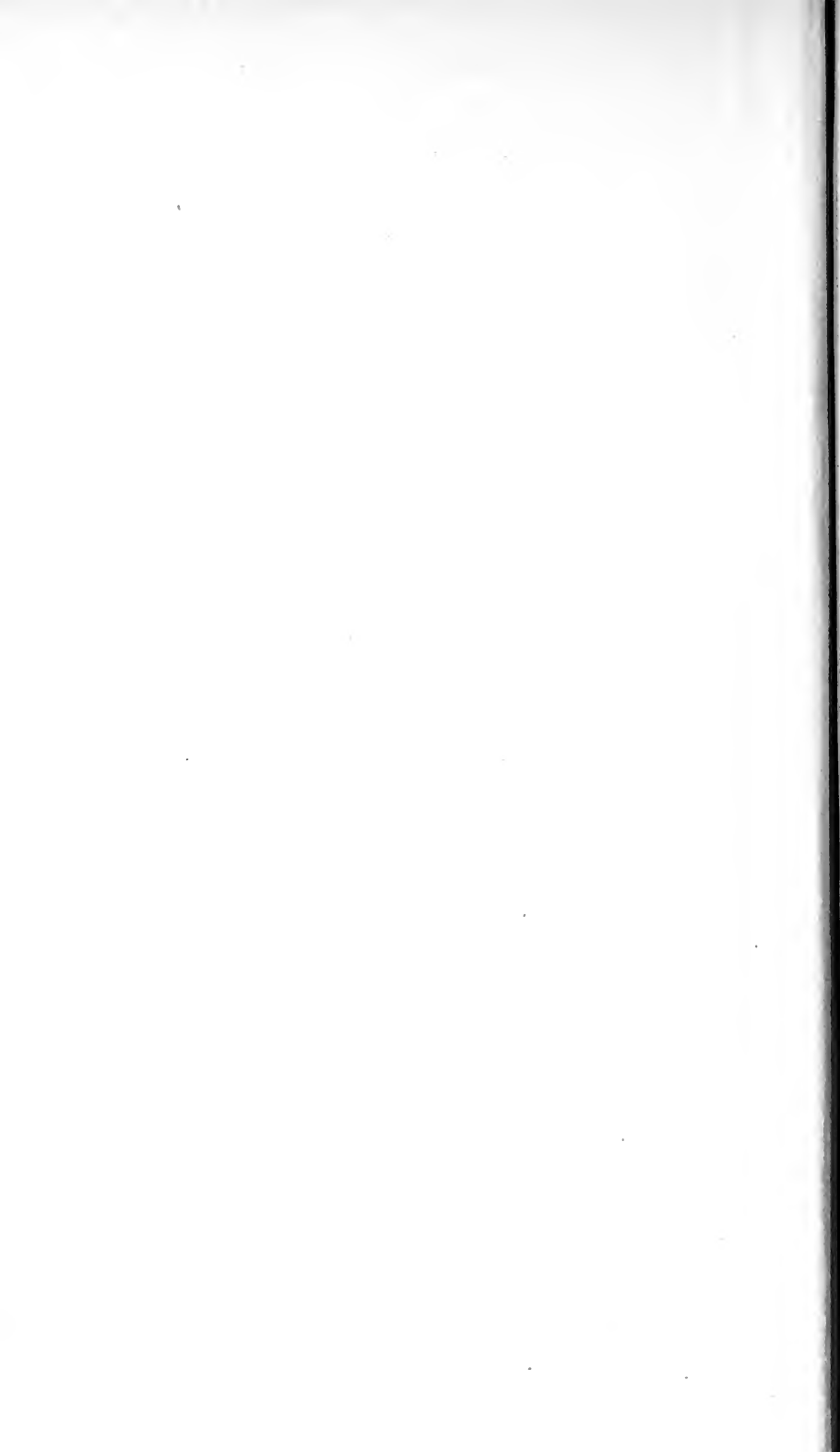
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# OFFICIAL AND PROVISIONAL METHODS OF ANALYSIS, ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS.

[Superseding Bulletins 46 and 65 on methods of analysis.]

## I. METHODS FOR THE ANALYSIS OF FERTILIZERS.<sup>a</sup>

[The word "water" whenever used throughout this bulletin means "distilled" water.]

### 1. Preparation of Sample.—Official.

Grind the sample to pass through a sieve having circular perforations 1 mm in diameter, and then thoroughly mix. Perform the grinding and sifting as rapidly as possible, to avoid loss or gain of moisture during the operation.

### 2. Moisture.—Official.

Heat 2 grams, or 5 grams if the samples be very coarse, for five hours in a water oven at the temperature of boiling water. In potash salts, sodium nitrate, and ammonium sulphate heat from 1 to 5 grams at about 130° C. until it ceases to lose weight. The loss in weight is considered as moisture.

### 3. Phosphoric Acid.—Official.

#### (a) GRAVIMETRIC METHOD.

##### (1) PREPARATION OF REAGENTS.

(a) *Ammonium citrate solution*.—Dissolve 370 grams of commercial citric acid in 1,500 cc of water; nearly neutralize with commercial ammonium hydroxid; cool; add ammonium hydroxid until exactly neutral (testing with saturated alcoholic solution of corallin), and dilute to a volume of 2 liters. Determine the specific gravity, which should be 1.09 at 20° C.

(b) *Optional method for ammonium citrate solution*.—To 370 grams of commercial citric acid add commercial ammonium hydroxid until nearly neutral; reduce the specific gravity to nearly 1.09 and make exactly neutral, testing as follows: Prepare a solution of fused calcium chlorid, 200 grams to the liter, and add four volumes of strong alcohol. Make the mixture exactly neutral, using a small amount of freshly prepared corallin solution as preliminary indicator, and test finally by withdrawing a portion, diluting with an equal volume of water, and testing with cochineal solution; 50 cc of this solution will precipitate the citric acid from 10 cc of the citrate solution. To 10 cc of the nearly neutral citrate solution add 50 cc of the alcoholic calcium chlorid solution, stir well, filter at once through a folded filter, dilute with an equal volume of water, and test the reaction with neutral solution of cochineal. If acid or alkaline, add ammonium hydroxid or citric acid, as the case may be,

<sup>a</sup> For special treatment of Thomas or basic slag, see Appendix, page 233.

mix, and test again, as before. Repeat this process until a neutral reaction is obtained. Add sufficient water to make the specific gravity 1.09 at 20° C.

(c) *Molybdate solution*.—Dissolve 100 grams of molybdic acid in 144 cc of ammonium hydroxid, specific gravity 0.90, and 271 cc of water; slowly and with constant stirring, pour the solution thus obtained into 489 cc of nitric acid (specific gravity 1.42), and 1,148 cc of water. Keep the mixture in a warm place for several days, or until a portion heated to 40° C. deposits no yellow precipitate of ammonium phosphomolybdate. Decant the solution from any sediment and preserve in glass-stoppered vessels.

(d) *Ammonium nitrate solution*.—Dissolve 200 grams of commercial ammonium nitrate in enough water to make the volume of the solution 2 liters.

(e) *Magnesia mixture*.—Dissolve 22 grams of recently ignited calcined magnesia in dilute hydrochloric acid, avoiding an excess of the latter. Add a little calcined magnesia in excess, and boil a few minutes to precipitate iron, alumina, and phosphoric acid; filter; add 280 grams of ammonium chlorid, 261 cc of ammonium hydroxid of specific gravity 0.90, and water enough to make the volume 2 liters. Instead of the solution of 22 grams of calcined magnesia, 110 grams of crystallized magnesium chlorid ( $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ ) may be used.

(f) *Dilute ammonium hydroxid for washing*.—This solution should contain 2.5 per cent of ammonia ( $\text{NH}_3$ ).

(g) *Magnesium nitrate solution*.—Dissolve 320 grams of calcined magnesia in nitric acid, avoiding an excess of the latter; then add a little calcined magnesia in excess; boil; filter from the excess of magnesia, ferric oxid, etc., and dilute with water to 2 liters.

## (2) TOTAL PHOSPHORIC ACID.

(a) *Methods of making solution*.—Treat 2 grams of the sample by one of the methods given below. After solution, cool, dilute to 200 or 250 cc, mix, and pour on a dry filter.

(a<sub>1</sub>) Ignite and dissolve in hydrochloric acid.

(a<sub>2</sub>) Evaporate with 5 cc of magnesium nitrate, ignite, and dissolve in hydrochloric acid.

(a<sub>3</sub>) Boil with from 20 to 30 cc of strong sulphuric acid, adding from 2 to 4 grams of sodium or potassium nitrate at the beginning of the digestion and a small quantity after the solution has become nearly colorless, or adding the nitrate in small portions from time to time. A Kjeldahl flask marked at 250 cc is recommended. After the solution is colorless add 150 cc of water and boil for a few minutes, cool, and make up to mark.

(a<sub>4</sub>) Digest with strong sulphuric acid and such other reagents as are used in either the plain or modified Kjeldahl or Gunning method for estimating nitrogen. Do not add any potassium permanganate, but after the solution has become colorless add about 100 cc of water and boil for a few minutes, cool, and make up a convenient volume; 2.5 grams of substance and a digestion flask marked at 250 cc are recommended.

(a<sub>5</sub>) Dissolve in 30 cc of concentrated nitric acid and a small quantity of hydrochloric acid and boil until organic matter is destroyed.

(a<sub>6</sub>) Add 30 cc of concentrated hydrochloric acid, heat, and add cautiously, in small quantities at a time, about 0.5 gram of finely pulverized potassium chlorate to destroy organic matter.

(a<sub>7</sub>) Dissolve in from 15 to 30 cc of strong hydrochloric acid and from 3 to 10 cc of nitric acid. This method is recommended for fertilizers containing much iron or aluminum phosphate.

(b) *Determination.*—Take an aliquot portion of the solution prepared above corresponding to 0.25 gram, 0.50 gram, or 1 gram. neutralize with ammonium hydroxid, and clear with a few drops of nitric acid. In case hydrochloric or sulphuric acid has been used as solvent, add about 15 grams of dry ammonium nitrate or a solution containing that amount. To the hot solution add 50 cc of molybdate solution for every decigram of phosphoric acid ( $P_2O_5$ ) that is present. Digest at about  $65^\circ$  C. for an hour, filter, and wash with cold water or, preferably, ammonium nitrate solution. Test the filtrate for phosphoric acid by renewed digestion and addition of more molybdate solution. Dissolve the precipitate on the filter with ammonium hydroxid and hot water and wash into a beaker to a bulk of not more than 100 cc. Nearly neutralize with hydrochloric acid, cool, and add magnesia mixture from a burette; add slowly (about 1 drop per second), stirring vigorously. After fifteen minutes add 12 cc of ammonium hydroxid solution, specific gravity 0.90. Let stand for some time—two hours is usually enough; filter, wash with 2.5 per cent ammonia ( $NH_3$ ) until practically free from chlorids; ignite to whiteness or to a grayish white and weigh.

### (3) WATER-SOLUBLE PHOSPHORIC ACID.

Place 2 grams of the sample on a 9-cm filter, wash with successive small portions of water, allowing each portion to pass through before adding more, until the filtrate measures about 250 cc. If the filtrate be turbid, add a little nitric acid. Make up to any convenient definite volume, mix well, use an aliquot, and proceed as under total phosphoric acid.

### (4) CITRATE-INSOLUBLE PHOSPHORIC ACID.

(a) *Determination in acidulated samples.*—Heat 100 cc of strictly neutral ammonium citrate solution of 1.09 specific gravity to  $65^\circ$  C. in a flask placed in a warm-water bath, keeping the flask loosely stoppered to prevent evaporation. When the citrate solution in the flask has reached  $65^\circ$  C., drop into it the filter containing the washed residue from the water-soluble phosphoric acid determination, close tightly with a smooth rubber stopper, and shake violently until the filter paper is reduced to a pulp. Place the flask in the bath and maintain it at such a temperature that the contents of the flask will stand at exactly  $65^\circ$  C. Shake the flask every five minutes. At the expiration of exactly thirty minutes from the time the filter and residue are introduced remove the flask from the bath and immediately filter the contents as rapidly as possible. Wash thoroughly with water at  $65^\circ$  C. (a) Transfer the filter and its contents to a crucible, ignite until all organic matter is destroyed, add from 10 to 15 cc of strong hydrochloric acid, and digest until all phosphate is dissolved; or (b) return the filter with contents to the digestion flask, add from 30 to 35 cc strong nitric acid, from 5 to 10 cc strong hydrochloric acid, and boil until all phosphate is dissolved. Dilute the solution to 200 cc. If desired, the filter and its contents may be treated according to methods ( $a_2$ ), ( $a_3$ ), or ( $a_4$ ) under total phosphoric acid. Mix well, filter through a dry filter; take a definite portion of the filtrate and proceed as under total phosphoric acid.

(b) *Determination in nonacidulated samples.*—In case a determination of citrate-insoluble phosphoric acid is required in nonacidulated samples it is to be made by treating 2 grams of the phosphatic material without previous washing with water, precisely in the way above described, except that in case the substance contains much animal matter (bone, fish, etc.), the residue insoluble in ammonium citrate is to be treated by any one of the processes described under total phosphoric acid ( $a_2$ ), ( $a_3$ ), or ( $a_4$ ), page 2.

## (5) CITRATE-SOLUBLE PHOSPHORIC ACID.

The sum of the water-soluble and citrate-insoluble subtracted from the total gives the citrate-soluble phosphoric acid.

## (b) OPTIONAL VOLUMETRIC METHOD.

## (1) PREPARATION OF REAGENTS.

(a) *Molybdate solution*.—To 100 cc of molybdate solution, prepared as directed on page 2, add 5 cc of nitric acid, specific gravity 1.42. This solution should be filtered each time before using.

(b) *Standard sodium or potassium hydroxide solution*.—Dilute 323.81 cc of normal alkali, which has been freed from carbonates, to 1 liter. One hundred cubic centimeters of the solution should neutralize 32.38 cc of normal acid; 1 cc is equal to 1 mg of  $P_2O_5$  (1 per cent of  $P_2O_5$  on a basis of 0.1 gram of substance).

(c) *Standard acid solution*.—The strength of this solution is the same as, or one-half of, the standard alkali solution, and is determined by titrating against that solution, using phenolphthalein as indicator. Any mineral acid may be used.

(d) *Phenolphthalein solution*.—Dissolve one gram of phenolphthalein in 100 cc of alcohol.

## (2) TOTAL PHOSPHORIC ACID.

(a) *Methods of making solution*.—Dissolve according to methods ( $a_2$ ), ( $a_3$ ), ( $a_6$ ), or ( $a_7$ ), (page 2), preferably by ( $a_6$ ), when these acids are a suitable solvent, and dilute to 200 cc with water.

(b) *Determination*.—( $b_1$ ) For percentages of 5 or below use an aliquot corresponding to 0.4 gram of substance, for percentages between 5 and 20 use an aliquot corresponding to 0.2 gram of substance, and for percentages above 20 use an aliquot corresponding to 0.1 gram of substance. Add from 5 to 10 cc of nitric acid, depending on the method of solution (or the equivalent in ammonium nitrate), nearly neutralize with ammonium hydroxid, dilute to from 75 to 100 cc, heat in water bath to from 60° to 65° C., and for percentages below 5 add from 20 to 25 cc of freshly filtered molybdate solution. For percentages between 5 and 20 add from 30 to 35 cc of molybdate solution; stir, let stand about 15 minutes, filter *at once*, wash once or twice with water by decantation, using from 25 to 30 cc each time, agitating the precipitate thoroughly and allowing to settle; transfer to filter and wash with cold water until two fillings of the filter do not greatly diminish the color produced with phenolphthalein by one drop of the standard alkali. Transfer precipitate and filter to beaker or precipitating vessel, dissolve in small excess of standard alkali, add a few drops of phenolphthalein solution, and titrate with standard acid.

( $b_2$ ) Proceed as directed in ( $b_1$ ) with this exception: Heat in a water bath at 45° to 50° C., add the molybdate solution, and allow to remain in the bath with occasional stirring for 30 minutes.

( $b_3$ ) Proceed as in ( $b_1$ ) to the point where the solution is ready to place in the water bath. Then cool solution to room temperature, add molybdate solution at the rate of 75 cc for each decigram of phosphoric acid present, place the stoppered flask containing the solution in a shaking apparatus and shake for 30 minutes at room temperature, filter at once, wash, and titrate as in preceding method.

## (3) WATER-SOLUBLE PHOSPHORIC ACID.

Dissolve according to directions given under "Gravimetric method," (3), page 3. To an aliquot portion of the solution corresponding to 0.2 or 0.4

gram, add 10 cc of concentrated nitric acid and ammonium hydroxid until a slight permanent precipitate is formed, dilute to 60 cc, and proceed as under the preceding method (2) ( $b_1$ ), page 4.

(4) CITRATE-INSOLUBLE PHOSPHORIC ACID.

Make the solution according to the directions given under "Gravimetric method" (4), page 3, and determine the phosphoric acid in an aliquot corresponding to 0.4 gram, as directed in (2) ( $b_1$ ), page 4.

(5) CITRATE-SOLUBLE PHOSPHORIC ACID.

The sum of the water-soluble and citrate-insoluble subtracted from the total gives the citrate-soluble phosphoric acid.

4. Nitrogen.

(a) KJELDAHL METHOD.—OFFICIAL.

(Not applicable in the presence of nitrates.)<sup>a</sup>

(1) PREPARATION OF REAGENTS.

(a) *Standard acid solution.*—( $a_1$ ) Standard hydrochloric acid, the absolute strength of which has been determined by precipitating with silver nitrate and weighing the silver chlorid as follows:

By means of a preliminary test with silver-nitrate solution, to be measured from a burette, with excess of calcium carbonate to neutralize free acid and potassium chromate as indicator, determine exactly the amount of nitrate required to precipitate all the hydrochloric acid. To a measured and also to a weighed portion of the standard acid add from a burette one drop more of silver-nitrate solution than is required to precipitate the hydrochloric acid. Heat to boiling, cover from the light, and allow to stand until the precipitate is granular. Then wash with hot water through a Gooch crucible, testing the filtrate to prove excess of silver nitrate. Dry the silver chlorid at 140° to 150° C.

( $a_2$ ) *Standard sulphuric acid.*—The absolute strength of the acid must be determined by precipitation with barium chlorid. For ordinary work half-normal acid is recommended. For work in determining very small amounts of nitrogen tenth-normal acid is recommended. In titrating mineral acids against ammonium hydroxid solution use cochineal as indicator.

(b) *Standard alkali solution.*—The strength of this solution relative to the acid must be accurately determined; tenth-normal solution is recommended.

(c) *Sulphuric acid.*—The sulphuric acid used should have a specific gravity of 1.84 and be free from nitrates and also from ammonium sulphate.

(d) *Metallic mercury, or mercuric oxid.*—If mercuric oxid is used, it should be prepared in the wet way, but not from mercuric nitrate.

(e) *Potassium permanganate.*—This reagent is used in a finely pulverized state.

<sup>a</sup> Mix 5 grams of the fertilizer with 25 cc of hot water and filter. To a portion of this solution add two volumes of concentrated sulphuric acid, free from nitric acid and oxids of nitrogen, and allow the mixture to cool. Add cautiously a few drops of a concentrated solution of ferrous sulphate, so that the fluids do not mix. If nitrates are present the junction shows at first a purple, afterwards a brown color, or if only a very minute quantity be present, a reddish color. To another portion of the solution add 1 cc of a dilute solution of nitrate of soda (3 grams to 300 cc) and test as before to determine whether sufficient sulphuric acid were added in the first test. (U. S. Dept. Agr., Division of Chemistry, Bul. 49, p. 19.)

(f) *Granulated zinc or pumice stone*.—One of these reagents is added to the contents of the distillation flasks, when found necessary, in order to prevent bumping.

(g) *Potassium sulphid solution*.—A solution of 40 grams of commercial potassium sulphid in 1 liter of water.

(h) *Sodium hydroxid solution*.—A saturated solution of sodium hydroxid free from nitrates.

(i) *Indicator*.—A solution of cochineal is prepared by digesting and frequently agitating 3 grams of pulverized cochineal in a mixture of 50 cc of strong alcohol and 200 cc of distilled water for a day or two at ordinary temperatures. The filtered solution is employed as indicator.

## (2) APPARATUS.

(a) *Kjeldahl flasks for both digestion and distillation*.—These are flasks having a total capacity of about 550 cc, made of hard, moderately thick, and well-annealed glass. When used for distillation the flasks are fitted with rubber stoppers and bulb tubes, as given under distillation flasks.

(b) *Kjeldahl digestion flasks*.—These are pear-shape, round bottom flasks, made of hard, moderately thick, well-annealed glass, having a total capacity of about 250 cc. They are 22 cm long and have a maximum diameter of 6 cm, tapering gradually to a long neck, which is 2 cm in diameter at the narrowest part and flared a little at the edge.

(c) *Distillation flasks*.—For distillation a flask of ordinary shape, of about 550 cc capacity, may be used. It is fitted with a rubber stopper and with a bulb tube above to prevent the possibility of sodium hydrate being carried over mechanically during distillation. The bulbs may be about 3 cm in diameter, the tubes being of the same diameter as the condenser and cut off obliquely at the lower end, which is fastened to the condenser by a rubber tube.

## (3) DETERMINATION.

Place from 0.7 to 3.5 grams of the substance to be analyzed, according to its proportion of nitrogen, in a digestion flask (2) with approximately 0.7 gram of mercuric oxid, or its equivalent in metallic mercury, and from 20 to 30 cc of sulphuric acid. Place the flask in an inclined position and heat below the boiling point of the acid for from five to fifteen minutes, or until frothing has ceased. (A small piece of paraffin may be added to prevent extreme foaming.) Then raise the heat until the acid boils briskly and digest for a time after the mixture is colorless or nearly so, or until oxidation is complete. (With some materials, as leather scrap, cheese, milk products, etc., it is necessary to digest for several hours.) Remove the flask from the flame, hold it upright, and while still hot drop potassium permanganate in carefully and in small quantities at a time until, after shaking, the liquid remains of a green or purple color.

After cooling dilute with about 200 cc of water, add a few pieces of granulated zinc or pumice stone when this is necessary in order to keep the contents of the flask from bumping, and 25 cc of potassium sulphid solution with shaking. Next add 50 cc of the soda solution, or sufficient to make the reaction strongly alkaline, pouring it down the side of the flask so that it does not mix at once with the acid solution. Connect the flask with the condenser, mix the contents by shaking, and distil until all ammonia has passed over into the standard acid. The first 150 cc of the distillate will generally contain all the ammonia. This operation usually requires from forty minutes to one hour and a half. The distillate is then titrated with standard alkali.

The use of mercuric oxid in this operation greatly shortens the time necessary for digestion, which is rarely over an hour and a half in case of substances most

difficult to oxidize, and is more commonly less than an hour. In most instances the use of potassium permanganate is quite unnecessary, but it is believed that in exceptional cases it is required for complete oxidation, and in view of the uncertainty it is always used. The potassium sulphid removes all the mercury from the solution, and so prevents the formation of mercur-ammonium compounds which are not completely decomposed by the sodium hydroxid. The addition of zinc gives rise to an evolution of hydrogen and prevents violent bumping. Previous to use the reagents should be tested by a blank experiment with sugar, which will partially reduce any nitrates present that might otherwise escape notice.

(b) GUNNING METHOD.—OFFICIAL.

(Not applicable in the presence of nitrates.<sup>a</sup>)

(1) PREPARATION OF REAGENTS.

(a) *Potassium sulphate*.—This reagent should be pulverized before using.

The other standard solutions and reagents used are the same as those described under the Kjeldahl method (p. 5).

(2) APPARATUS.

The apparatus used is the same as that employed in the Kjeldahl method (p. 6).

(3) DETERMINATION.

Place the substance to be analyzed in a digestion flask, employing from 0.7 to 3.5 grams, according to its proportion of nitrogen. Add 10 grams of powdered potassium sulphate and from 15 to 25 cc. (ordinarily about 20 cc) of sulphuric acid. Conduct the digestion as in the Kjeldahl process, starting with a temperature below boiling point and increasing the heat gradually until frothing ceases. Digest for a time after the mixture is colorless or nearly so, or until oxidation is complete. Do not add either potassium permanganate or potassium sulphid. Dilute, neutralize, distil, and titrate as in the Kjeldahl method. In neutralizing it is convenient to add a few drops of phenolphthalein indicator, by which one can tell when the acid is completely neutralized, remembering that the pink color, which indicates an alkaline reaction, is destroyed by a considerable excess of strong fixed alkali.

(C) KJELDAHL METHOD MODIFIED TO INCLUDE THE NITROGEN OF NITRATES.—OFFICIAL.

(1) PREPARATION OF REAGENTS.

Besides the reagents given under the Kjeldahl method, there will be needed—

(a) *Zinc dust*.—This should be an impalpable powder. Granulated zinc or zinc filings will not answer.

(b) *Sodium thiosulphate*.

(c) *Commercial salicylic acid*.

(2) APPARATUS.

The apparatus used is the same as in the Kjeldahl method, page 6 (2).

(3) DETERMINATION.

Place from 0.7 to 3.5 grams of the substance to be analyzed in a Kjeldahl digestion flask, add 30 cc of sulphuric acid containing 1 gram of salicylic acid,

<sup>a</sup> See page 5 footnote, for qualitative method for determining the presence of nitrates.

and shake until thoroughly mixed, then add 5 grams of crystallized sodium thiosulphate; or add to the substance 30 cc of sulphuric acid containing 2 grams of salicylic acid, then add gradually 2 grams of zinc dust, shaking the contents of the flask at the same time. Finally, place the flask on the stand for holding the digestion flasks, where it is heated over a low flame until all danger from frothing has passed. The heat is then raised until the acid boils briskly and the boiling continued until white fumes no longer escape from the flask. This requires about five or ten minutes. Add approximately 0.7 gram of mercuric oxid, or its equivalent in metallic mercury, and continue the boiling until the liquid in the flask is colorless, or nearly so. In case the contents of the flask are likely to become solid before this point is reached, add 10 cc more of sulphuric acid. Complete the oxidation with a little potassium permanganate in the usual way and proceed with the distillation as described in the Kjeldahl method. The reagents should be tested by blank experiments.

(d) GUNNING METHOD MODIFIED TO INCLUDE THE NITROGEN OF NITRATES.—  
OFFICIAL.

(1) PREPARATION OF REAGENTS.

Besides the reagents given under the Gunning method there will be needed—

- (a) *Sodium thiosulphate.*
- (b) *Commercial salicylic acid.*

(2) APPARATUS.

The apparatus used is the same as that given under the Kjeldahl method, page 6 (2).

(3) DETERMINATION.

In a digestion flask place from 0.7 to 3.5 grams of the substance to be analyzed, according to the amount of nitrogen present. Add from 30 to 35 cc of salicylic acid mixture, namely, 30 cc sulphuric acid to 1 gram of salicylic acid; shake until thoroughly mixed, and allow to stand from 5 to 10 minutes, with frequent shaking. Add 5 grams of sodium thiosulphate and heat the solution for 5 minutes; cool; add 10 grams of potassium sulphate and heat. This reduces the danger of foaming. Heat very gently until foaming ceases, then heat strongly until nearly colorless. Dilute, neutralize, and distil as in the Gunning method.

(e) ABSOLUTE OR CUPRIC OXID METHOD.—OFFICIAL.

(Applicable to all nitrogen determinations.)

(1) PREPARATION OF REAGENTS.

- (a) *Coarse cupric oxid.*—To be ignited and cooled before using.
- (b) *Fine cupric oxid.*—Prepared by grinding ordinary cupric oxid.
- (c) *Metallic copper.*—Granulated copper, or fine copper gauze, heated and cooled in a current of hydrogen.
- (d) *Sodium bicarbonate*, free from organic matter.
- (e) *Caustic potash solution.*—A supersaturated solution of caustic potash in hot water.

(2) APPARATUS.

- (a) Combustion tube of best hard Bohemian glass, about 66 cm long and 12.7 mm internal diameter.



- (b) Azotometer of at least 100 cc capacity, accurately calibrated.
- (c) Sprengel mercury air pump.
- (d) Small paper scoop made from stiff writing paper.

### (3) DETERMINATION.

Use from 1 to 2 grams of ordinary commercial fertilizers. In the case of highly nitrogenized substances, the amount to be used must be regulated by the amount of nitrogen estimated to be present. Fill the tube as follows: (1) About 5 cm of coarse cupric oxid. (2) Place on the small paper scoop enough of the fine cupric oxid to fill about 10 cm of the tube, after having been mixed with the substance to be analyzed; pour on this the substance, rinsing the watch glass with a little of the fine oxid, and mix thoroughly with a spatula; pour into the tube, rinsing the scoop with a little fine oxid. (3) About 30 cm of coarse cupric oxid. (4) About 7 cm of metallic copper. (5) About 6 cm of coarse cupric oxid. (6) A small plug of asbestos. (7) From 0.8 to 1 gram of sodium bicarbonate. (8) A large loose plug of asbestos. Place the tube in the furnace, leaving about 2.5 cm of it projecting; connect with the pump by a rubber stopper smeared with glycerol, taking care to make the connection perfectly tight.

Exhaust the air from the tube by means of the pump. When a vacuum has been obtained, allow the flow of mercury to continue; light the gas under that part of the tube containing the metallic copper, the anterior layer of cupric oxid (see (5) above), and the sodium bicarbonate. As soon as the vacuum is destroyed and the apparatus filled with carbon dioxid, shut off the flow of mercury and at once introduce the delivery tube of the pump into the receiving arm of the azotometer just below the surface of the mercury seal so that the escaping bubbles will pass into the air and not into the tube, thus avoiding the useless saturation of the caustic potash solution.

When the flow of carbon dioxid has very nearly or completely ceased, pass the delivery tube down into the receiving arm, so that the bubbles will escape into the azotometer. Light the gas under the 30 cm layer of oxid, heat gently for a few moments, to drive out any moisture that may be present, and bring to a red heat. Heat gradually the mixture of substance and oxid, lighting one jet at a time. Avoid a too rapid evolution of bubbles, which should be allowed to escape at the rate of about one per second, or a little faster.

When the jets under the mixture have all been turned on, light the gas under the layer of oxid at the end of the tube. When the evolution of gas has ceased, turn out all the lights except those under the metallic copper and anterior layer of oxid, and allow to cool for a few moments. Exhaust with the pump and remove the azotometer before the flow of mercury is stopped. Break the connection of the tube with the pump, stop the flow of mercury, and extinguish the lights. Allow the azotometer to stand for at least an hour, or cool with a stream of water until a permanent volume and temperature have been reached.

Adjust accurately the level of the potassium hydroxid solution in the bulb to that in the azotometer; note the volume of gas, temperature, and height of barometer; make calculation as usual or read results from tables.

### (f) MAGNESIUM OXID METHOD.—OFFICIAL.

(Applicable only to the determination of ammonia.)

Place from 0.7 to 3.5 grams of the substance to be analyzed, according to the proportion of ammonia present, in a distillation flask (p. 6, (2), (a)), with about

200 cc of water and 5 grams or more of magnesium oxid free of carbon dioxid. Then connect the flask with a condenser and distil 100 cc of the liquid into standard acid. Titrate the residual acid as in the Kjeldahl method, page 6.

(g) ULSCH METHOD, MODIFIED BY STREET.—OFFICIAL.

(Applicable to all nitric and ammoniacal nitrogen determinations.)

Place 1 gram of the sample in a half-liter flask. Add about 30 cc of water and 2 to 3 grams of reduced iron, and after standing sufficiently long to insure solution of the soluble nitrates and ammonia salts add 10 cc of a mixture of strong sulphuric acid with an equal volume of water; shake thoroughly and allow to stand for a short time until the violence of the reaction has moderated. Place a long-stemmed funnel in the neck of the flask to prevent mechanical loss. Heat the solution slowly, boiling it for five minutes, and cool. Add about 100 cc of water, a little paraffin, and from 7 to 10 grams of magnesium oxid, free or nearly free from carbonates. Connect with a condenser, such as is used in the Kjeldahl method, and boil the mixture for forty minutes, nearly to dryness; collect the ammonia in a known amount of standard acid, and titrate in the usual manner. The nitrogen obtained represents the nitrates, plus the ammonia salts, contained in the sample.

In the analysis of nitrate salts proceed as above, except that 25 cc of the nitrate solution, equivalent to 0.25 gram of sample, are employed with 5 grams of reduced iron. After boiling add 75 cc of water and an excess of sodium hydrate and complete the determination as above.

(h) ZINC-IRON METHOD.—OFFICIAL.

(Applicable to the determination of nitric and ammoniacal nitrogen.)

Dissolve 10 grams of the sample in 500 cc of water. Of this solution place 25 cc, corresponding to one-half gram, in a distillation flask of about 400 cc capacity, add 120 cc of water, also about 5 grams of well-washed and dried zinc dust, and an equal weight of reduced iron. To the solution add 80 cc of sodium hydrate of 32° B. Then connect the flask with the condensing apparatus and conduct the distillation synchronously with the reduction, collecting the ammonia in carefully standardized acid. Continue the distillation for one or two hours, or until 100 cc have been distilled and titrate the resulting distillate as in the Kjeldahl method, page 6.

(i) NEUTRAL-PERMANGANATE METHOD.—PROVISIONAL.

(For the determination of available organic nitrogen.)

Into a 300 cc low-form Griffin beaker weigh 2 grams of the sample if from a mixed fertilizer; if from concentrated material, use a quantity containing approximately 0.075 gram of nitrogen (sample containing material that has been treated with acid should be washed on a 9 cm S. S. No. 595 filter to 200 cc and transferred, filter and all, to a beaker). Digest this with 125 cc of permanganate of potash solution (16 grams of pure potassium permanganate to 1,000 cc water) in a steam or hot-water bath for thirty minutes. Have the beaker let down well into the steam or hot water and keep closed with cover glass, stirring twice at intervals of ten minutes with a glass rod. At the expiration of the time remove from bath, add 100 cc of cold water, and filter through a heavy 15 cm folded filter. Wash with cold water, small quantities at a time, till total filtrate amounts to 400 cc. Dry and determine nitrogen in residue by Kjeldahl method.

## (j) ALKALINE-PERMANGANATE METHOD.—PROVISIONAL.

(For the determination of available organic nitrogen.)

Weigh out an amount of sample containing 0.045 gram of nitrogen and transfer to a 600 cc distillation flask. After connecting with a condenser, to which a receiver containing standard acid has been attached, digest below the boiling point with 100 cc of alkaline permanganate solution (16 grams of potassium permanganate and 150 grams of sodium hydroxid dissolved in water and made to volume of 1 liter) for thirty minutes. Then boil until 85 cc of the distillate is obtained. If the material shows a tendency to adhere to the sides of the flask, an occasional gentle rotation is necessary during distillation.

## 5. Potash.

## (a) LINDO-GLADDING METHOD.—OFFICIAL.

## (1) PREPARATION OF REAGENTS.

(a) *Ammonium chlorid solution*.—Dissolve 100 grams of ammonium chlorid in 500 cc of water, add from 5 to 10 grams of pulverized potassium-platinic chlorid, and shake at intervals for six or eight hours. Allow the mixture to settle overnight and filter. The residue may be used for the preparation of a fresh supply.

(b) *Platinum solution*.—The platinum solution used contains 1 gram of metallic platinum (2.1 grams of  $\text{H}_2\text{PtCl}_6$ ) in every 10 cc.

## (2) METHODS OF MAKING SOLUTION.

(a) *With potash salts and mixed fertilizers*.—Boil 10 grams of the sample with 300 cc of water thirty minutes. In the case of mixed fertilizers, add to the hot solution a slight excess of ammonium hydroxid and then sufficient powdered ammonium oxalate to precipitate all the lime present. Cool, dilute to 500 cc, mix, and pass through a dry filter. In the case of muriate and sulphate of potash, sulphate of potash and magnesia, and kainit, dissolve and dilute to 500 cc without the addition of ammonium hydroxid and ammonium oxalate.

(b) *With organic compounds*.—When it is desired to determine the total amount of potash in organic substances, such as cottonseed meal, tobacco stems, etc., saturate 10 grams with strong sulphuric acid and ignite in a muffle at a low red heat to destroy organic matter. Add a little strong hydrochloric acid, warm slightly in order to loosen the mass from the dish, and proceed as directed under (3) (a) below.

## (3) DETERMINATION.

(a) *In mixed fertilizers*.—Evaporate 50 cc of the solution made according to (2), corresponding to 1 gram of the sample, nearly to dryness, add 1 cc of dilute sulphuric acid (1 to 1), evaporate to dryness, and ignite to whiteness. As all the potash is in the form of sulphate, no loss need be apprehended by volatilization of potash, and a full red heat must be maintained until the residue is perfectly white. Dissolve the residue in hot water, using at least 20 cc for each decigram of potassium oxid, add a few drops of hydrochloric acid, and platinum solution in excess. Evaporate on a water bath to a thick paste and treat the residue with 80 per cent alcohol, sp. gr. 0.8645, avoiding the absorption of ammonia. Wash the precipitate thoroughly with 80 per cent alcohol both by decantation and on the filter, continuing the washing after the filtrate is colorless. Wash finally with 10 cc of the ammonium chlorid solution (1) (a) to remove impurities from the precipitate and repeat this

washing five or six times. Wash again thoroughly with 80 per cent alcohol and dry the precipitate for thirty minutes at 100° C. The precipitate should be perfectly soluble in water.

(b) *Muriate of potash*.—Dilute 25 cc of the solution, prepared according to (2) (a), with 25 cc of water, acidify with a few drops of hydrochloric acid, add 10 cc of platinum solution and evaporate to a thick paste. Treat the residue as under (3) (a).

(c) *Sulphate of potash; sulphate of potash and magnesia, and kainit*.—Dilute 25 cc of the solution, prepared according to (2) (a), with 25 cc of water, acidify with a few drops of hydrochloric acid and add 15 cc of platinum solution. Evaporate the mixture and proceed as directed under (3) (a), except that 25 cc portions of ammonium chlorid solution should be used.

(d) *Water-soluble potash in wood ashes and cotton hull ashes*.—Use above method, making the solution according to (2) (a), and pay special attention to the last sentence of (3) (a).

#### (b) OPTIONAL METHOD.—OFFICIAL.<sup>a</sup>

##### (1) PREPARATION OF REAGENT.

*Platinum solution*.—The platinum solution used is the same as that described under the Lindo-Gladding method.

##### (2) METHOD OF MAKING SOLUTION.

The solution is prepared as directed under the Lindo-Gladding method, omitting in all cases the addition of ammonium hydroxid and ammonium oxalate.

##### (3) DETERMINATION.

Dilute 25 cc of the solution made as directed under (2) (50 cc if less than 10 per cent of potassium oxid be present) to 150 cc, heat to 100° C., and add, drop by drop, with constant stirring, a slight excess of barium chlorid solution. Without filtering, add in the same manner barium hydroxid in slight excess. Filter while hot and wash until the precipitate is free from chlorids. Add to the filtrate 1 cc of strong ammonium hydrate, and then a saturated solution of ammonium carbonate until the excess of barium is precipitated. Heat and add, in fine powder, 0.5 gram of pure oxalic acid or 0.75 gram of ammonium oxalate. Filter and wash free from chlorids, evaporate the filtrate to dryness in a platinum dish, and ignite carefully over the free flame, below a red heat, until all volatile matter is driven off. Digest the residue with hot water, filter through a small filter and dilute the filtrate, if necessary, so that for each decigram of potassium oxid there will be at least 20 cc of liquid. Acidify with a few drops of hydrochloric acid and add platinum solution in excess. Evaporate on a water bath to a thick paste and treat the residue with 80 per cent alcohol, sp. gr. 0.8645, both by decantation and after collecting on a Gooch or other form of filter. Dry for thirty minutes at 100° C. and weigh.

It is desirable, if there be an appearance of foreign matter in the double salt, that it should be washed according to the previous method with several portions of 10 cc each of ammonium chlorid solution.

##### (c) FACTORS.

For the conversion of potassium platinic chlorid to potassium chlorid use the factor 0.3071; to potassium sulphate, 0.3589; and to potassium oxid, 0.1941.

<sup>a</sup> The Lindo-Gladding method is preferable in the presence of soluble sulphates.

## II. METHODS FOR THE ANALYSIS OF SOILS.

### 1. Directions for Taking Samples.

#### (a) METHOD 1.—OFFICIAL.

Remove surface accumulations of decaying leaves, etc., and take samples with a soil tube or auger to the desired depth. If the tract to be studied is not of uniform character, divide into smaller tracts, that each may be uniform, and from such tracts take five or six representative samples to the depth of 6 inches, or to the change between the surface soil and the subsoil, in case such change occurs between the depth of 6 and 12 inches. In no case is the sample to be taken to a greater depth than 12 inches. If the surface soil extend to a greater depth, a separate sample below the depth of 12 inches is to be obtained. If the surface soil extend to a depth of less than 6 inches, and the difference between it and the subsoil is unusually great, a separate sample of the surface soil should be secured, besides the one to the depth of 6 inches. Mix the samples of each depth thoroughly and take subsamples of 2 to 4 pounds, drying the latter in a well-aired, cool place.

The depth to which the sample of subsoil should be taken will depend on circumstances. It is always necessary to know what constitutes the foundation of a soil to the depth of 3 feet at least, since the question of drainage, resistance to drought, etc., will depend essentially upon the nature of the substratum. But in ordinary cases 10 or 12 inches of subsoil will be sufficient for the purposes of examination in the laboratory. The sample should be obtained in other respects precisely like that of the surface soil, while that of the material underlying this subsoil may be taken with less exactness, perhaps at some ditch or other easily accessible point. Mix and subsample as above. The sampling should be done preferably when the soil is reasonably dry.

It is recommended that the weight of a given volume of the soil as it lies in the field be taken for calculating the percentage results obtained by analysis to pounds per given area of the soil.

#### (b) METHOD 2.—PROVISIONAL.

Remove surface accumulations of decaying leaves, etc., and take samples with a soil tube or auger to the desired depth. All samples of soils taken for analysis should be composite and should be composed of representative samples taken from at least five different places in the field sampled, each individual sample to be a column of uniform soil extending through the stratum sampled.

One composite sample should be taken from each important and distinctly different soil stratum to a depth of 40 inches, or 1 meter, including a composite sample from the arable stratum, or plowed soil, usually about 6 inches or 15 cm deep.

If the plow line and the subsoil line coincide, and the subsoil is a fairly uniform stratum to the depth of 40 inches, then only two composite samples need be taken, one of the arable soil and one of the subsoil. But if the subsoil line is lower than the plow line and not below 40 inches, then both strata below the

arable soil should be sampled, which would make three composite samples from the field—one from the surface or arable soil, one from the subsurface soil—that is, from the stratum between the plow line and the true subsoil line, and one from the true subsoil. Dry the samples in a well-aired, cool place.

## 2. Preparation of Sample.—Official.

Prepare the laboratory sample by putting it through a 1 mm sieve, using a rubber-tipped pestle to rub up the lumps. Weigh and discard the detritus. Keep the sample in a cool place and stopper to prevent change in condition. Use a 3 mm sieve for preparing that portion of the sample intended for determinations requiring 100 grams or more of soil.

## 3. Moisture.—Official.

Dry 2 or more grams in a tared platinum dish for 5 hours at the temperature of boiling water; cover the dish, cool in a desiccator, and weigh. Repeat the heating, cooling, and weighing at intervals of 2 hours, until the material ceases to lose weight. The loss of weight is reported as moisture. Weigh rapidly, to avoid absorption of moisture from the air.

## 4. Volatile Matter.—Official.

Heat the dish and dry soil from the above determination to full redness, occasionally stirring, until all organic matter is burned away. If the soil contain appreciable quantities of carbonates it is moistened, after cooling, with a few drops of a saturated solution of ammonium carbonate, dried and heated to dull redness to expel ammonium salts, cooled in the desiccator, and weighed. The loss in weight represents the organic matter, water of combination, salts of ammonium, etc. (See Appendix, p. 234, for Total Organic Carbon.)

## 5. Strong Acid Digestion of the Soil.

### (a) METHOD OF PREPARING SOIL SOLUTION.—OFFICIAL.

Digest a quantity equivalent to 10 grams of moisture free soil (this facilitates the subsequent calculations). Make the digestion preferably in an Erlenmeyer flask of nonsoluble glass of 200 or 300 cc capacity. The flask should have a ground-glass stopper terminating in a reflux tube 20 inches or more in length. A rubber stopper carrying a tube may be substituted if glass-stoppered flasks are not available. Use 100 cc of hydrochloric acid of a constant boiling point (sp. gr. 1.115), made approximately by diluting 1,350 cc of ordinary acid (sp. gr. 1.20), with 1,000 cc of water. Digest continuously for 10 hours on a steam or water bath, shaking the flask every hour. After settling, decant the solution into a porcelain dish or hard glass beaker. Very small quantities of the sediment passing over will do no harm. Wash the insoluble residue onto a filter with hot water and continue the washing until free from chlorids, adding the washings to the original solution for evaporation. Oxidize the organic matter present in the solution with a few drops of nitric acid and evaporate to dryness on a water bath. Take up with hot water and a few cubic centimeters of hydrochloric acid and again evaporate to complete dryness. When the final evaporation is complete and the dish cooled, add a few drops of strong hydrochloric acid, sufficient only to saturate the residue. Add 10 to 20 cc of water, warm on the bath to secure complete solution, and filter, washing until free from chlorids. Again evaporate this solution to dryness to render insoluble any silica that may yet be in solution, and treat as above. The filtrate

constitutes the acid extract freed of soluble silica, and is made up to a definite volume (250 or 500 cc) and designated as solution A.

Combine the two filters and the main residue and after drying ignite, preferably in a small dish over a Bunsen flame for an hour or more, then complete by igniting over a blast until it ceases to lose weight. Weigh as the insoluble residue.

(b) FERRIC AND ALUMINIC OXIDS AND PHOSPHATES, COLLECTIVELY.—OFFICIAL.

To an aliquot of solution A (50 or 100 cc, according to the probable amount of iron present) add ammonium hydroxid, drop by drop, until the precipitate formed requires several seconds to dissolve, thus leaving the solution but faintly acid. Heat nearly to the boiling point, add sufficient ammonium hydroxid to precipitate all of the iron, alumina, etc. Allow the covered beaker to boil for about one minute, remove, and if no ammonia is given off (detect by smelling), more is added drop by drop until it can be detected. Do not allow the precipitate to settle, but stir and pour onto the filter. Wash immediately with hot water, using a fine jet which is played around the edge of the precipitate, thus cutting it free from the paper in order to produce rapid filtration. Wash the precipitate several times and return it to the original beaker, dissolve with a few drops of hydrochloric acid and warm. Reprecipitate the iron, alumina, and phosphoric acid with ammonium hydroxid as above and wash until free from chlorids. The filtrate is designated as Solution B.

Dry the precipitate, remove it from the filter, and ignite over a Bunsen flame, the filter being incinerated separately and added to the precipitate. Then ignite to bright redness, cool in a desiccator and weigh as ferric oxid ( $\text{Fe}_2\text{O}_3$ ), alumina ( $\text{Al}_2\text{O}_3$ ), and phosphorus pentoxid ( $\text{P}_2\text{O}_5$ ). Transfer this residue to a flask and digest with several cubic centimeters of sulphuric acid (1 to 4), heating to accelerate solution. When solution is complete reduce with zinc and estimate ferric oxid with a standard solution of permanganate.

In lieu of the above, evaporate 50 or 100 cc of solution A with the addition of 10 cc of sulphuric acid until all hydrochloric acid is expelled, dilute with water, reduce with zinc, and estimate ferric oxid with a standard solution of permanganate.

The ferric oxid, together with the phosphorus pentoxid (to be determined later), subtracted from the collective weights of ferric oxid, alumina, and phosphorus pentoxid, gives alumina.

(c) MANGANESE.—OFFICIAL.

Concentrate Solution B to about 50 cc, cool, and make alkaline with ammonium hydroxid, add bromin water until the solution is colored, and heat to boiling in a covered beaker; again cool, and repeat the addition of ammonium hydroxid, bromin water and boiling. If manganese be present, slightly acidify the solution with acetic acid, immediately filter, and wash with hot water. Dry the precipitate and ignite over a Bunsen flame and weigh as mangano-manganic oxid ( $\text{Mn}_3\text{O}_4$ ). The filtrate from this, or if there is no precipitate the original solution, becomes Solution C.

(d) CALCIUM.—OFFICIAL.

Evaporate Solution C to about 50 cc, make slightly alkaline with ammonium hydroxid, and add, while still hot, ammonium oxalate solution, drop by drop, so long as any precipitate is produced, adding a few cubic centimeters in excess to convert the magnesium also into oxalate. Heat to boiling, allow to stand for 3 hours or longer, decant the clear solution on a filter, pour from 15 to 20 cc

of hot distilled water on the precipitate, and again decant the clear solution onto the filter. Dissolve the precipitate in the beaker with a few drops of hydrochloric acid, add a little water, and reprecipitate, boiling hot, by adding ammonium hydroxid, to a slight alkalinity, and a little ammonium oxalate solution; allow to stand as before and filter through the same filter; transfer the precipitate to the filter and wash it free from chlorids with hot water; dry, ignite the precipitate over the blast lamp until it ceases to lose weight, and weigh as calcium oxid. The filtrate and washings become Solution D.

(e) MAGNESIUM.—OFFICIAL.

Evaporate Solution D on the water bath to dryness and carefully heat to expel ammonium salts. Take up the residue, with 20 or 25 cc hot water and about 5 cc hydrochloric acid, filter, and wash. Concentrate to about 50 cc, cool and add sufficient acid sodium phosphate to precipitate the magnesium; then add gradually ammonium hydroxid, with constant stirring, until the solution is distinctly alkaline. Test with acid sodium phosphate to be sure that sufficient has been added. Allow to stand one-half hour, then add gradually 10 cc of strong ammonium hydroxid, cover closely to prevent escape of ammonia, and let stand in the cold. Filter after 12 hours, wash the precipitate free from chlorids, using 2.5 per cent ammonia water, dry, burn at first at a moderate heat, then ignite intensely, and weigh as magnesium-pyro-phosphate ( $\text{Mg}_2\text{P}_2\text{O}_7$ ).

(f) PHOSPHORIC ACID. (See also Appendix, p. 234.)

(1) GRAVIMETRIC METHOD.—OFFICIAL.

Evaporate 100 or 200 cc of Solution A to about 25 or 30 cc, neutralize with ammonium hydroxid and add about 10 cc additional, neutralize the excess of ammonium hydroxid with nitric acid, gradually add at once about 20 cc of molybdate solution <sup>a</sup> ("I. Fertilizers," (1) (c), p. 2) and place the beaker in a water bath at a temperature of 40° to 60° C. When the precipitate has settled sufficiently, draw out with a pipette about 5 cc of the clear liquid and test by allowing it to run into 5 cc of warm molybdate solution. If any precipitate be produced, return the test liquid to the main portion, add more molybdate solution, and repeat the operation until all the phosphoric acid is precipitated. After standing several hours at a temperature not above 60° C., filter off the ammonium phosphomolybdate. Wash the precipitate thoroughly with cold water, dissolve with ammonium hydroxid, and determine the phosphoric acid as magnesium pyrophosphate, as directed under total phosphoric acid in fertilizers, page 3.

(2) VOLUMETRIC METHOD.—PROVISIONAL.

Proceed as in the gravimetric method (1) until all the phosphoric acid is precipitated and then finish the determination as follows:

After standing for 3 hours at a temperature not above 60° C., filter on a small filter paper or on a gooch crucible and wash with cold water until two fillings of the filter do not greatly diminish the color produced with phenolphthalein by 1 drop of standard alkali. Return the filter and precipitate to the same beaker used for precipitating the phosphomolybdate, dissolve the yellow precipitate in standard sodium or potassium hydroxid, add a few drops of phenolphthalein solution and titrate excess of alkali with standard acid; 1 cc of the standard alkali should be made to equal 0.0005 gram of phosphoric acid ( $\text{P}_2\text{O}_5$ ).

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<sup>a</sup> It is better to pour the solution of phosphate into the molybdate solution.



## (g) SULPHURIC ACID.—OFFICIAL.

Evaporate 100 or 200 cc of Solution A nearly to dryness on a water bath to expel the excess of acid; then add 50 cc of distilled water; heat to boiling and add drop by drop a 10 per cent barium chlorid solution until no further precipitation occurs. Continue the boiling for about 5 minutes; allow to stand for 5 hours or longer in a warm place, pour the liquid on a tared gooch or on an ashless filter, treat the precipitate with from 15 to 20 cc of boiling water, transfer to the filter and wash with boiling water until the filtrate is free from chlorids. Dry the filter, ignite over a Bunsen burner, and weigh as barium sulphate, which multiplied by 0.34293 gives the sulphur trioxid.

## (h) POTASSIUM AND SODIUM.

## (1) OFFICIAL METHOD.

Treat the filtrate from the sulphuric acid determination (g) with ammonium hydroxid exactly as in 5 (b), page 15. Evaporate the filtrate and washings to dryness, heat below redness until ammonium salts are expelled, dissolve in hot water, add 5 cc of barium hydroxid solution, and heat to boiling; let settle for a few minutes, and test a little of the clear liquid with more barium hydroxid solution to be sure that enough has been added. When no further precipitate is produced, filter and wash thoroughly with hot water. Heat the filtrate to boiling, add ammonium hydroxid and ammonium carbonate to complete precipitation of the barium, calcium, etc., let stand a short time on the water bath, filter, and wash the precipitate thoroughly with hot water; evaporate filtrate and washings to complete dryness, expel ammonium salts by heat below redness, take up with a little hot water, add a few drops of ammonium hydroxid, a drop or two of ammonium carbonate, and a few drops of ammonium oxalate; let stand a few minutes on the water bath, set aside for a few hours, filter, evaporate to complete dryness on the water bath, and heat to dull redness until all ammonium salts are expelled and the residue is nearly or quite white. Dissolve in a minimum amount of water, filter into a tared platinum dish, add a few drops of hydrochloric acid, evaporate to dryness on the water bath, heat to dull redness, cool in a desiccator, and weigh as potassium and sodium chlorids. Repeat heating until constant weight is obtained. Dissolve in a small amount of water; if any residue remains, the separation must be repeated until the residue of potassium and sodium chlorids is entirely soluble. Dissolve the residue with water, add an excess of platinum chlorid, and proceed as directed under "I. Fertilizers," (3) (a), page 11.

Or, instead of the foregoing, evaporate to dryness a fresh aliquot of solution A, redissolve in water, treat directly with barium hydroxid, and from this point proceed as above directed.

## (2) PROVISIONAL METHOD.

Proceed as in the official method (1) through "let stand a short time on the water bath" and complete as follows:

Filter into a beaker, add a drop or two of hydrochloric acid and 1 cc of ammonium sulphate (75 grams to 1 liter), digest several hours on water bath, and filter into a tared platinum dish. Evaporate to complete dryness, heat to full redness, add 1 gram of powdered ammonium carbonate, expel by heating, cool, and weigh the sulphates of sodium and potassium. Determine potassium in the usual manner.

### 6. Acid-Insoluble Materials.—Official.

The residue from acid soluble may be analyzed by the usual methods for silicates. If it is desired to determine the silica soluble in alkalis, the residue must be dried at 100° C. and an aliquot removed before ignition for treatment with sodium carbonate solution, as described under determination of inorganic plant constituents (d), page 22. Another aliquot, or the remainder of the residue, is ignited and weighed.

### 7. Total Alkalies.—Official.

Determine in a separate portion of the soil by J. Lawrence Smith's method, given in Crookes's Select Methods, second edition, pages 28–40, and Principles and Practice of Agricultural Analysis, 1894, volume I, pages 378–381, or, preferably, determine by this method the alkalies in the insoluble residue from "5. (a) Method of preparing soil solution," page 14, and add the amount obtained from the hydrochloric-acid solution.

### 8. The More Active Forms of Phosphoric Acid in Soils.—Provisional.

#### (a) SOLUTIONS REQUIRED.

Prepare a large stock solution of normal hydrochloric acid by titrating against a standard potassium hydroxid solution containing little or no carbonate, using phenolphthalein as the indicator. Also prepare a fifth-normal solution of hydrochloric acid.

#### (b) DETERMINATION.

Digest 10 grams of air-dried soil, in a stoppered flask, with 100 cc of fifth-normal hydrochloric acid, for exactly five hours in a water bath kept at a temperature of 40° C. Filter the solution through a dry paper, cool to the room temperature, and titrate 20 cc of the filtrate with standard carbonate-free potassium hydroxid solution, using phenolphthalein as the indicator. From the data thus secured calculate the exact number of cubic centimeters of normal acid of the stock solution and of water to make exactly one or two liters of acid of fifth-normal strength after allowing for the amount neutralized by the amount of soil to be used in the following procedure.

Place 200 grams of the air-dried soil in a large, dry, glass-stoppered bottle and add exactly 2,000 cc of fifth-normal hydrochloric acid corrected for neutralization as above described. In the case of soils known to be rich in available phosphoric acid 100 grams of soil and 1,000 cc of acid will be sufficient. Place the bottle in a large water bath and keep at a temperature of 40° C. for exactly five hours, shaking thoroughly each half hour. At the end of the digestion shake contents of bottle well and pour quickly upon a large, dry, ribbed filter of two thicknesses of paper and of sufficient size to receive the entire contents of the bottle. The filtrate is to be received in a dry vessel and the solution poured back through the paper until entirely clear. Evaporate 1,000 cc of the filtrate if 200 grams of soil be used, or 500 cc if 100 grams be employed, to dryness in a porcelain dish, after adding a few cubic centimeters of nitric acid to oxidize organic matter, etc., moisten the residue with hydrochloric acid, take up with water, and filter into a flask of about 500 cc capacity. Add 15 grams of ammonium nitrate in solution, then strong ammonium hydroxid until a permanent precipitate forms, and then concentrated nitric acid until the precipitate dissolves. Dilute to about 100 cc, if less than that volume, place a ther-

mometer in the flask, and heat to exactly 85° C. Add 75 cc of recently prepared molybdate solution, digest in a water bath at 80° C. for 15 minutes, with occasional shaking, remove from the bath and allow to stand at least 10 minutes before filtering. Continue the determination in the usual way.

#### 9. Lithium, Cæsium, and Rubidium.—Official.

The salts of these elements are occasionally found in very small amounts in soils. Their agricultural uses are still in question, and their amount is too small to admit of quantitative estimation. A qualitative examination may be made by the spectroscope with the water-soluble materials evaporated to dryness and dissolved with two or three drops of hydrochloric acid or with the alkaline chlorids separated as in 5 (h), page 17, or "7. Total Alkalies," page 18.

#### 10. Total Nitrogen.—Official.

Place from 7 to 14 grams of the soil in a Kjeldahl digesting flask, with 30 cc of strong sulphuric acid, or more if necessary, and 0.7 gram of mercuric oxid (or its equivalent in metallic mercury), and boil for several hours. Oxidize the residue with potassium permanganate in the usual way. After cooling half fill the flask with water, shake it vigorously, allow the heavy matters to subside, and pour the supernatant liquid into a flask of about 1,000 cc capacity. Repeat this operation until the ammonium sulphate is practically all removed and the distillation flask is a little more than half full; distil the ammonia in the usual manner. If the sample be known to contain a considerable amount of nitrate, use method "4. (c)," page 7, under analysis of fertilizers.

#### 11. Carbon Dioxid.—Official.

Determine as under analysis of inorganic plant constituents, "2. (c)," page 21, using from 5 to 10 grams of the sample.

#### 12. Humus.—Official.

Place 10 grams of the sample in a gooch crucible, extract with 1 per cent hydrochloric acid until the filtrate gives no precipitate with ammonium hydroxid and ammonium oxalate, and remove the acid by washing with water. Wash the contents of the crucible (including the asbestos filter) into a glass-stoppered cylinder, with 500 cc of 4 per cent ammonium hydroxid, and allow to remain, with occasional shaking, for 24 hours. During this time the cylinder is inclined as much as possible without bringing the contents in contact with the stopper, thus allowing the soil to settle on the side of the cylinder and exposing a very large surface to the action of the ammonium hydroxid. Place the cylinder in a vertical position and leave for 12 hours, to allow the sediment to settle. Filter the supernatant liquid (the filtrate must be perfectly clear), evaporate an aliquot, dry at 100° C., and weigh. Then ignite the residue and again weigh. Calculate the humus from the difference in weights between the dried and ignited residues.

#### 13. Humus Nitrogen.—Official.

Digest the soil with 2 per cent hydrochloric acid and wash as nearly free of acid as possible with distilled water. Extract the humus with a 3 per cent solution of sodium hydroxid and determine nitrogen in the extract in the usual way.

## 14. Soil Acidity.—Provisional.

Place 100 grams of soil in a 400 cc wide-mouthed bottle, add 250 cc of normal potassium nitrate solution, stopper, and shake continuously for three hours in a shaking machine, or every five minutes by hand. Let stand over night. Draw off 125 cc of the clear supernatant liquid, boil ten minutes to expel carbon dioxid, cool, and titrate with standard sodium hydroxid solution. One cubic centimeter is equivalent to 4 mg of calcium carbonate, using phenolphthalein as indicator.

The acids and acid salts of the soil are difficultly soluble in water, but by treating with a salt solution, such as sodium chlorid or potassium nitrate, a double decomposition takes place, rendering them soluble. An equilibrium is reached, however, before this reaction runs to an end, and if, after having drawn off 125 cc to titrate, 125 cc of fresh potassium nitrate are added to the bottle and the bottle again shaken for three hours, 125 cc drawn off will give a titration which is more than one-half of the first. By continuing this process until the last 125 cc shows practically no acidity, a series of titrations is obtained the sum of which represents the total acidity of the 100 grams of soil. It has been found by working with a number of different soils that as an average the sum of such a series is 2.5 times the first titration.

Consequently when the sodium hydroxid is made up so that 1 cc is equivalent to 4 mg of calcium carbonate and 125 cc (which represents 50 grams of soil) are titrated, each 0.1 cc required to neutralize corresponds to 1 mg of calcium carbonate required by the 100 grams of soil, or to 0.001 per cent of calcium carbonate required by the soil tested.

## 15. Statement of Results.—Official.

Calculate all results of soil analysis as per cent of the soil dried to constant weight in the water oven (see "3. Moisture," p. 14) and state in the following order:

Insoluble matter	-----
Soluble silica	-----
Potash ( $K_2O$ )	-----
Soda ( $Na_2O$ )	-----
Lime ( $CaO$ )	-----
Magnesia ( $MgO$ )	-----
Manganese oxid ( $Mn_2O_4$ )	-----
Ferric oxid ( $Fe_2O_3$ )	-----
Alumina ( $Al_2O_3$ )	-----
Phosphorus pentoxid ( $P_2O_5$ )	-----
Sulphur trioxid ( $SO_3$ )	-----
Carbon dioxid ( $CO_2$ )	-----
Volatile matter	-----
-----	
Total	-----
Humus	-----
Ash	-----
Phosphorus pentoxid	-----
Silica	-----
Nitrogen (organic)	-----
Hygroscopic moisture	-----
Moisture absorbed at $t^\circ$	-----

### III. METHODS FOR THE ANALYSIS OF INORGANIC PLANT CONSTITUENTS.<sup>a</sup>

(For provisional combustion method see Appendix, p. 236.)

#### 1. Preparation of Sample.—Official.

Thoroughly cleanse the material from all foreign matter, especially from adhering soil. Grind, and preserve the sample in carefully stoppered bottles.

#### 2. Carbon-Free Ash.—Official.

##### (a) PREPARATION OF STANDARD CALCIUM ACETATE.

(1) Dissolve 20 grams of chemically pure calcium carbonate in chemically pure acetic acid and dilute to 1 liter. To standardize this evaporate 20 cc in a platinum dish, ignite gently, then strongly, to constant weight. The dish must be weighed quickly. This procedure gives the calcium oxid in 20 cc.

(2) An alternative method is to dissolve marble in hydrochloric acid, evaporate, and dry to render silica insoluble, dissolve with water and a little acid, and precipitate iron and aluminum in the usual way. Then precipitate the calcium with ammonium hydroxid and ammonium oxalate in hot solution, wash well, dry, ignite, and weigh. Dissolve in acetic acid and dilute so that 100 cc contain 1.1 grams of calcium oxid. It is best to test the purity of this reagent by making blank determinations.

##### (b) PREPARATION OF ASH.

(For optional official method without the use of calcium acetate see Appendix, p. 238.)

Molsten 10 to 20 grams of substance with 40 cc of calcium acetate, dry on a water bath, and ignite, gently at first, then more vigorously. The quantity of calcium acetate used should be sufficient to prevent fusion of the ash. Some form of apparatus must be used to prevent volatilization, either Shuttleworth's<sup>b</sup> or Tucker's<sup>c</sup>, or an ordinary platinum dish may be used, fitted with a cover, like that described by Wislicenus.<sup>d</sup> The weight of the ash must be corrected for lime, carbon dioxid, and carbon.

##### (c) CARBON DIOXID.

Using the ash prepared in (b), liberate the carbon dioxid with hydrochloric acid in any of the usual forms of apparatus, determining the carbon dioxid evolved either by increase of weight of potash bulbs or loss of weight of the apparatus. The former method is preferred.

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<sup>a</sup> Neither provisional nor official methods for the determination of iron and aluminum have been adopted, but such determinations should be made.

<sup>b</sup> Exper. Stat. Rec., 11: 304.

<sup>c</sup> Ibid., 506.

<sup>d</sup> Zts. anal. Chem., 1901, 40: 441.

## (d) CARBON, SAND, AND SILICA.

Transfer the residue from the carbon dioxid determination to a beaker or evaporating dish; evaporate to dryness and thoroughly dry and pulverize to render silica insoluble. Moisten the dry residue with from 5 to 10 cc of hydrochloric acid, take up with about 50 cc of water, allow to stand on the water bath for a few minutes, filter through a parchment-paper filter (S. and S. "hardened" filters), and thoroughly wash. Make the solution and washings up to 250 cc or other convenient volume and preserve for analysis. Designate as Solution A.

(1) Wash the residue from the filter (which may be used again) into a platinum dish and boil about 5 minutes with approximately 20 cc of a saturated solution of pure sodium carbonate, add a few drops of pure sodium hydroxid solution, allow the solids to settle, and decant the liquor through a tared gooch. Boil the residue in the dish with sodium carbonate solution and decant as before. Repeat the process a third time, after which bring the residue upon the filter and thoroughly wash, first with hot water, then with a little dilute hydrochloric acid, and finally with hot water until free from chlorids. Dry the gooch and contents to constant weight at 110° C., thus determining the combined weight of carbon and sand. After incineration the loss in weight gives the carbon. It is advisable to examine the residue under the microscope to ascertain if it is really sand. The alkaline filtrate and washings are united, acidified with hydrochloric acid, evaporated to dryness, and the silica separated and determined in the usual way.

(2) Instead of determining directly the silica dissolved by the sodium carbonate solution, as described above, another portion of the ash may be treated as in 2 (c) and (d), and the residue of silica, sand, and carbon filtered on an ordinary filter, washed, burned, and weighed, giving the combined weight of silica and sand, from which the weight of sand found in 2 (d) is to be deducted to obtain the silica. It is inadmissible to separate the soluble silica from the residue after ignition.

Subtract carbon, carbon dioxid, and calcium oxid, added in the form of calcium acetate, from the ash, and calculate results as carbon-free ash.

### 3. Manganese, Calcium, and Magnesium.—Official.

To an aliquot of Solution A, corresponding to 0.5 to 2 grams of ash, add a quantity of pure ferric chlorid solution, more than equivalent to the phosphoric acid which may be present, neutralize with ammonium hydroxid, dissolve the precipitate in a very slight excess of hydrochloric acid, add 1 or 2 grams of sodium acetate, and boil one or two minutes, filter at once, and wash with boiling water. Dissolve the precipitate in hydrochloric acid and reprecipitate as above. Evaporate the filtrate and washings to about 50 cc, and determine manganese, calcium, and magnesium as in the analysis of soils ((c), (d), and (e), p. 15). The quantity of calcium found must be corrected for the calcium added.

### 4. Phosphoric Acid.—Official.

(1) In an aliquot of the hydrochloric-acid solution, corresponding to from 0.2 to 1 gram of ash, determine phosphoric acid by either of the methods for total phosphoric acid given under fertilizers ((a) (2) or (b) (2), pp. 2 and 4).

(2) The determination can also be made in the plant substance as in (a) (2), page 2, for determining total phosphoric acid in fertilizers, using sufficient material to give from 0.2 to 1 gram of ash in the aliquot of the solution used.

### 5. Sulphuric Acid, Sodium, and Potassium.—Official.

Heat to boiling an aliquot of Solution A, corresponding to from 0.5 to 1 gram of ash, add barium chlorid solution in small quantities until no further precipitation is produced, and proceed as under soils (5 (g) and (h), p. 17).

### 6. Chlorin.—Official.

Determine as silver chlorid, either gravimetrically or by one of the standard volumetric processes (as the Volhard method given below), in a nitric acid or aqueous solution of the ash.

#### VOLHARD METHOD OF DETERMINING CHLORIN.

##### (a) SOLUTIONS REQUIRED.

(1) *Silver nitrate*.—Prepare a decinormal solution in the usual way.

(2) *Ammonium or potassium thiocyanate*.—Standardize a decinormal solution by means of the standard silver solution.

(3) *Ferric indicator*.—Prepare a saturated solution of iron alum.

(4) *Pure nitric acid*.—This reagent must not contain any of the lower oxids of nitrogen.

##### (b) DETERMINATION.

Dissolve the material (in this case ash) in nitric acid and filter. Add a known volume of the tenth-normal silver nitrate to the filtrate until an excess is present. Stir well, filter, and wash the silver chlorid precipitate thoroughly. To the filtrate and washings add 5 cc of the ferric indicator and a few cubic centimeters of nitric acid. Titrate the excess of silver with the tenth-normal thiocyanate solution until a permanent light-brown color appears. From the amount of tenth-normal silver nitrate originally used subtract the excess as shown by the thiocyanate solution to obtain the amount used by the chlorin.

### 7. Potassium in Plants.—Official.

Determine potash as directed under fertilizers for potash in organic compounds ((2) (b), p. 11), using sufficient plant material to get from 0.5 to 1 gram of ash in the aliquot of the solution used for the potash determination.

### 8. Sulphur in Plants—Peroxid Method.—Provisional.

Place from 1.5 to 2.5 grams of material in a nickel crucible of about 100 cc capacity and moisten with approximately 2 cc of water. Mix thoroughly, using a nickel or platinum rod. Add 5 grams of pure anhydrous sodium carbonate and mix. Add pure sodium peroxid, small amounts (approximately 0.50 gram) at a time, thoroughly mixing the charge, after each addition. Continue adding the peroxid until the mixture becomes nearly dry and quite granular, requiring usually about 5 grams of peroxid. Place the crucible over a low alcohol flame (or other flame free from sulphur) and carefully heat with occasional stirring until contents are fused. (Should the material ignite, the determination is worthless.) After fusion remove the crucible, allow to cool somewhat, and cover the hardened mass with peroxid to a depth of about 0.5 cm. Heat gradually, and finally with full flame until complete fusion takes place, rotating

the crucible from time to time in order to bring any particles adhering to the sides into contact with the oxidizing material. Allow to remain over the lamp for ten minutes after fusion is complete. Cool somewhat. Place warm crucible and contents in a 600 cc beaker and carefully add about 100 cc of water. After violent action has ceased, wash material out of crucible, make slightly acid with hydrochloric acid (adding small portions at a time), transfer to a 500 cc flask, cool, and make to volume. Filter, and take a 200 cc aliquot for determination of sulphates by precipitating with barium chlorid in the usual manner.

#### 9. Chlorin in Plants.—Provisional.

Impregnate 5 grams of substance in a platinum dish with 20 cc of a 5 per cent solution of sodium carbonate, evaporate to dryness, and ignite as thoroughly as possible. Extract the residue with hot water, filter, and wash. Return to the platinum dish, ignite to an ash, dissolve in nitric acid, and determine chlorin by the Volhard method (p. 23).



## IV. METHODS FOR THE ANALYSIS OF INSECTICIDES AND FUNGICIDES.

### 1. General Directions.—Provisional.

Samples of Paris green, London purple, soft soap, copper carbonate, and tobacco extracts should be thoroughly mixed before analysis, taking care in the case of the first two substances that they are not further pulverized. Weigh large quantities of lye and potassium cyanid in weighing bottles and analyze aliquots of the water solutions.

### 2. Paris Green.

#### (a) MOISTURE,<sup>a</sup>—PROVISIONAL.

Dry from 1 to 2 grams at 105° to 110° C., and calculate the loss as moisture.

#### (b) TOTAL ARSENIOS OXID,<sup>b</sup> METHOD I (SMITH).—OFFICIAL.

##### (1) SOLUTIONS REQUIRED.

(a) *Starch solution*.—Use a starch solution which is prepared by boiling 2 grams of starch with 200 cc of distilled water for about 5 minutes.

(b) *Standard iodine solution*.—Prepare a standard iodine solution in the following manner: Dissolve 12.7 grams of powdered iodine in about 250 cc of water to which has been added about 25 grams of chemically pure potassium iodide, and make up the whole to a volume of 2 liters. To standardize this solution, weigh 1 gram of chemically pure dry arsenious oxide, transfer to a 250 cc flask by means of about 100 cc of a solution containing 2 grams of sodium hydroxide in each 100 cc, and boil until all arsenious oxide goes into solution. Make up to a volume of 250 cc and use 50 cc for analysis. Concentrate this portion of 50 cc, by boiling in a 250 cc flask, to half its volume and allow to cool to about 80° C. Add an equal volume of concentrated hydrochloric acid and 3 grams of potassium iodide, mix, and allow the whole to stand for 10 minutes (to reduce the arsenic oxide formed by boiling the alkaline arsenite to arsenious oxide). Dilute the solution with cold water and add an approximately tenth-normal solution of sodium thiosulphate, drop by drop, until the solution becomes exactly colorless. (The end point is easy to read without the aid of starch.) Make this solution slightly alkaline with dry sodium carbonate (using a drop of methyl orange to read the change) and then make slightly acid with hydrochloric acid, taking care that all lumps of sodium carbonate on the bottom are acted on by the hydrochloric acid. Add sodium bicarbonate in excess and run in the solution of iodine drop by drop, using starch water to read the end reaction. (Sometimes the solution becomes dark toward the end of the titration. This change must not be confused with the final dark-blue color given by the iodine starch.)

<sup>a</sup> J. Amer. Chem. Soc., 1900, 22 (9) : 568.

<sup>b</sup> Ibid., 1899, 21 (8) : 769.

From the number of cubic centimeters of iodine solution and the weight of arsenious oxide used determine the value of each cubic centimeter of iodine in terms of arsenious oxide.

(2) DETERMINATION.

Transfer 2 grams of Paris green to a 250 cc flask by means of about 100 cc of a 2 per cent sodium-hydroxide solution. Boil this mixture for 5 to 10 minutes, or until all the green particles have changed to red cuprous oxide, then cool it to room temperature and make the volume up to 250 cc. Filter the well-shaken liquid through a dry filter and use 50 cc portions for analysis. Conduct the analysis from this point as when standardizing the iodine solution.

(c) TOTAL ARSENIOS OXIDE, METHOD II (AVERY).—OPTIONAL OFFICIAL.

(1) SOLUTIONS REQUIRED.

(a) *Standard iodine solution*.—Prepare an iodine solution as in Method I (p. 25). To standardize this solution place 1 gram of dry chemically pure arsenious oxide in a 250 cc flask, and dissolve by boiling about 20 minutes with 5 grams of sodium bicarbonate and approximately 100 cc of water; cool; add hydrochloric acid until acid, and then sodium bicarbonate until alkaline; make up to the mark and titrate aliquots of 50 cc with iodine solution as in Method I.

(b) *Sodium acetate solution*.—Dissolve 12.5 grams of the crystallized salt in each 25 cc of water.

(c) *Sodium potassium tartrate solution*.—Dissolve from 2 to 3 grams of sodium potassium tartrate in each 50 cc of water.

(d) *Starch solution*.—Prepare a starch solution as in Method I.

(2) DETERMINATION.

Place 1 gram of Paris green in a 100 cc flask and boil for 5 minutes with 25 cc of the sodium acetate solution. Make to the mark, shake, and pass through a dry asbestos gooch filter. Use an aliquot of this filtrate for the determination of the soluble arsenious oxide by means of the iodine solution. Transfer the residue on the filter to a beaker, beat up with a little water, dissolve in concentrated hydrochloric acid added a drop at a time, then add 3 or 4 drops in excess. Transfer the whole to the 100 cc flask originally employed and analyze aliquots of from 20 to 40 cc. Add concentrated sodium carbonate solution, a drop at a time, to each of these aliquots until a slight permanent precipitate is formed. Dissolve this precipitate by adding 50 cc of the sodium potassium tartrate. Dilute to about 200 cc, add solid sodium bicarbonate and starch water, and titrate with standard iodine.

(d) TOTAL ARSENIOS OXIDE, METHOD III (AVERY-HAYWOOD).—OPTIONAL OFFICIAL.

(1) SOLUTIONS REQUIRED.

Prepare the same solutions as were required for Method II.

(2) DETERMINATION.

Boil 0.4 gram of the finely ground Paris green with 25 cc of the sodium acetate solution for from 5 to 10 minutes. Add concentrated hydrochloric acid, a drop at a time, until solution is effected (about 10 cc of the acid is usually necessary). Add concentrated sodium carbonate solution, a drop at a time, until

a slight precipitate appears, then proceed as directed in the last two sentences of Method II.

(e) SODIUM-ACETATE-SOLUBLE ARSENIOUS OXID.<sup>a</sup>—PROVISIONAL.

(1) SOLUTIONS REQUIRED.

Prepare the same solutions as are used in Method II for total arsenious oxid, with the exception of the sodium potassium tartrate solution.

(2) DETERMINATION.

Proceed as described in the first three sentences of Method II for total arsenious oxid, except that a paper filter is used instead of asbestos.

(f) WATER-SOLUBLE ARSENIOUS OXID.<sup>b</sup>—PROVISIONAL.

(1) SOLUTIONS REQUIRED.

Prepare starch and standard iodine solutions as described under Method II.

(2) DETERMINATION.

Treat 1 gram of Paris green in a large flask with 1,000 cc of water (previously boiled to expel carbon dioxide and then cooled to room temperature). Stopper the flask and shake eight times each day for ten days. At the end of this time filter the solution through a dry filter. Treat 200 cc of this filtrate with sodium bicarbonate and titrate with the iodine solution.

(g) TOTAL COPPER OXID, METHOD I (ELECTROLYTIC).—OFFICIAL.

Pour the cuprous oxid (obtained in Method I, page 26, for total arsenious oxid by boiling the Paris green with sodium hydroxid) on the filter and wash well with hot water, after an aliquot of the filtrate has been used for the determination of arsenious oxid. Then dissolve in hot dilute nitric acid and make up to a volume of 250 cc. Use 50 to 100 cc of this solution for the electrolytic determination of copper, as described on page 52, paragraph (2), under "VII. General Methods for the Analysis of Foods and Feeding Stuffs."

(h) TOTAL COPPER OXID,<sup>b</sup> METHOD II (THIOSULPHATE).—OPTIONAL OFFICIAL.

(1) SOLUTIONS REQUIRED.

Standard thiosulphate solution. Dissolve 24.8 grams of the crystallized salt in water and make up to 2 liters. Standardize this solution against chemically pure copper foil dissolved in nitric acid by the method of analysis given in the following paragraph:

(2) DETERMINATION.

Use an aliquot portion of the nitric-acid solution of copper oxid, employed in Method I for total copper oxid. Make it alkaline with sodium carbonate, then make *slightly* acid with acetic acid, dilute with water, and add about 3 or 4 grams of solid potassium iodid. When the potassium iodid is all dissolved by

<sup>a</sup> J. Amer. Chem. Soc., 1901, 23 (2) : 111.

<sup>b</sup> Ibid., 1900, 22 (9) : 568.

shaking, titrate the free iodine with thiosulphate, using starch as indicator toward the end of the reaction.

### 3. London Purple.

#### (a) MOISTURE.<sup>a</sup>—PROVISIONAL.

Dry from 1 to 2 grams for from 10 to 12 hours at a temperature of 105° to 110° C.

#### (b) TOTAL ARSENIOS OXID,<sup>a</sup> METHOD I (HAYWOOD).—PROVISIONAL.

##### (1) SOLUTIONS REQUIRED.

Prepare starch and iodine solutions by either of the methods given under "2. Paris green," pages 25 and 26.

##### (2) DETERMINATION.

Dissolve 2 grams of London purple in a mixture of about 80 cc of water and 20 cc of concentrated hydrochloric acid at a temperature of from 60° to 70° C.; filter and wash to a volume of 300 cc. Treat 100 cc of this solution with sodium bicarbonate in excess and make up to the mark in a 500 cc flask, using a few drops of ether to destroy the bubbles. Pass a portion through a dry filter, and to 250 cc add starch water, and titrate the solution with standard iodine to the appearance of a blue color. The result is the arsenious oxide, as such, in 50 cc of the original solution, or in 0.3333 gram of the original London purple.

#### (c) TOTAL ARSENIC OXID,<sup>a</sup> METHOD I (HAYWOOD).—PROVISIONAL.

##### (1) SOLUTIONS REQUIRED.

Use the same solutions as described above for total arsenious oxide (b) (1).

##### (2) DETERMINATION.

Heat 50 cc of the hydrochloric-acid solution of London purple, prepared by the preceding method, to 80° C. on the water bath, remove and add 50 cc of concentrated hydrochloric acid and 3 grams of potassium iodide. Allow the mixture to stand for at least 15 minutes, the arsenic acid thus being reduced to the arsenous condition and the iodine set free. Then rinse the solution into a large beaker, dilute well, and add twentieth-normal sodium thiosulphate, drop by drop, to eliminate the free iodine. The end point here is rather difficult to read on account of the very dark color of the solution, but with a little practice the chemist can determine it by proceeding as follows:

Run in the sodium thiosulphate a little at a time, occasionally withdrawing a drop of the solution and adding it to a drop of starch paste. This will, of course, give a blue color with the starch, which becomes fainter as the iodine is used up. Finally, when a drop of the solution gives only the slightest blue color with the starch, add a little starch paste directly to the whole solution and dissipate the blue color with a few drops of thiosulphate. With a little practice the chemist can in this way get the exact end point. Immediately make the solution alkaline with solid sodium carbonate. Again make it slightly acid with hydrochloric acid, taking care that all of the solid particles of the sodium carbonate on the bottom are neutralized by the acid, and finally make alkaline with sodium bicarbonate. Add starch paste and titrate with the standard iodine solution.

<sup>a</sup> J. Amer. Chem. Soc., 1900, 22 (12) : 800.

The end point is easily read if the beaker is placed on a white surface between the eye and the light and the iodine solution run in until a distinct purple color appears. The figure thus obtained gives the number of cubic centimeters of iodine corresponding to the total amount of arsenic in the solution expressed as arsenious oxide. Subtracting from this the number of cubic centimeters of iodine corresponding to the arsenious oxide in the previous method gives the number of cubic centimeters of iodine corresponding to the arsenic oxide in 0.3333 gram of the sample.

(d) TOTAL ARSENIOS OXIDE,<sup>a</sup> METHOD II (HAYWOOD-DAVIDSON).—PROVISIONAL

(Designed to eliminate part of coloring matter.)

(1) SOLUTIONS REQUIRED.

Use the same solutions as in Method I for total arsenious oxide.

(2) DETERMINATION.

Place 2 grams of London purple in a beaker and dissolve in about 80 cc of water and 20 cc of concentrated hydrochloric acid at a temperature of 60° to 70° C., cool and add sodium carbonate in slight excess, transfer to a 250-cc flask, bring to the mark, shake, and filter through a dry filter. Acidify 50 cc of the filtrate with hydrochloric acid and make alkaline with sodium bicarbonate. Titrate the amount of arsenious oxide present with the standard iodine solution.

(e) TOTAL ARSENIC OXIDE,<sup>b</sup> METHOD II (HAYWOOD-DAVIDSON).—PROVISIONAL

(Designed to eliminate part of coloring matter.)

(1) SOLUTIONS REQUIRED.

Use the same solutions as in Method I for total arsenious oxide.

(2) DETERMINATION.

Acidify 50 cc of the solution, prepared as directed in the preceding paragraph, with concentrated hydrochloric acid, heat to 80° C., add 50 cc more of hydrochloric acid and 3 grams of potassium iodide, and proceed as described in Method I for total arsenic oxide (page 28), beginning with the second sentence.

(f) WATER-SOLUBLE ARSENIOS OXIDE,<sup>b</sup>—PROVISIONAL

(1) SOLUTIONS REQUIRED.

Use the same solutions as in Method I for total arsenious oxide.

(2) DETERMINATION.

Extract 1 gram of London purple in a stoppered flask with 500 cc of cold carbon-dioxide-free water for seven days, shaking eight times each day. Filter through a dry filter, to 100 cc of filtrate add sodium bicarbonate, and titrate with standard iodine, using starch as indicator.

<sup>a</sup> U. S. Dept. Agr., Bureau of Chemistry, Bul. 81, p. 199.

<sup>b</sup> J. Amer. Chem. Soc., 1900, 22 (12) : 800.

(g) WATER-SOLUBLE ARSENIC OXID.<sup>a</sup>—PROVISIONAL.

## (1) SOLUTIONS REQUIRED.

Use the same solutions as in Method I for total arsenious oxid.

## (2) DETERMINATION.

Transfer an aliquot (about 200 cc) of the water extract from the determination of soluble arsenious oxid to a flask, make slightly alkaline with sodium hydroxid, and concentrate to about 25 cc. Remove the flask and allow it to cool to about 80° C., and add an equal volume of concentrated hydrochloric acid and 3 grams of potassium iodid. Allow it to stand 15 minutes, dilute, exactly use up the iodine set free with twentieth-normal thiosulphate (using starch if necessary), and neutralize the solution with sodium carbonate. Again make slightly acid with hydrochloric acid, taking care that all lumps of sodium carbonate are acted on, then make alkaline with an excess of sodium bicarbonate, and titrate with iodine, using starch as indicator. From this figure subtract the figure representing the amount of soluble arsenious oxid, and calculate the remainder as arsenic oxid.

## 4. Copper Carbonate.

## (a) COPPER OXID.—OFFICIAL.

## (1) SOLUTIONS REQUIRED.

Use the same solutions as described under the determination of total copper in Paris green (page 27).

## (2) DETERMINATION.

Dissolve a weighed quantity of the substance in dilute nitric acid and employ one of the methods of analysis given under the determination of total copper in Paris green (page 27).

## 5. Potassium Cyanid.

(a) CYANOGEN.<sup>b</sup>—OFFICIAL.

## (1) SOLUTIONS REQUIRED.

Prepare a twentieth-normal solution of silver nitrate.

## (2) DETERMINATION.

Weigh a large quantity of the sample in a weighing bottle, dissolve in water, and make up to a definite volume. To an aliquot add twentieth-normal silver nitrate, a drop at a time, with constant stirring, until one drop produces a permanent turbidity. In calculating the results, one equivalent of silver is equal to two equivalents of cyanogen, according to the following equation:



## 6. Soap.

## (a) GENERAL STATEMENT.—PROVISIONAL.

In most soaps, not considering resin soaps, it is necessary to know three constituents in order to judge of their value for spraying purposes, namely, mois-

<sup>a</sup> J. Amer. Chem. Soc., 1900, 22 (12): 800.

<sup>b</sup> Sutton's Volumetric Analysis, 5th ed., p. 200.

ture, total fatty matter, and total soda or potash. The moisture and alkali are usually determined and the total fatty matter approximately estimated by difference.

(b) **MOISTURE**<sup>a</sup> (MODIFIED METHOD OF BENEDIKT AND LEWKOWITSCH).—PROVISIONAL.

Tare accurately a 100 cc beaker, the bottom of which is covered about one-half inch deep with recently ignited, perfectly dry sand, and in which is a small glass rod. Weigh in the beaker about 5 grams of the sample; add 25 cc of alcohol or more if necessary, and dissolve the soap in the alcohol by constant stirring on the water bath. Evaporate the alcohol and finally dry in an oven at 110° C. until the weight is constant. A few precautions should be taken which are not mentioned in the above method, namely: If the soap is hard the 5 grams should be cut off in very thin strips so that it will dissolve more readily in the alcohol; also most samples of soap never come to a constant weight on drying, but gain or lose indefinitely. It is, therefore, best to heat the soap at 110° C. until it is nearly dry and weigh, then return the soap to the oven and dry another half hour. Continue this alternate drying and weighing until the weight changes only a few milligrams during the course of a half hour's drying.

(c) **TOTAL ALKALI**.<sup>b</sup>—PROVISIONAL.

Dissolve a weighed quantity of the soap in water; decompose with hydrochloric acid, filter off the water from the fat, and wash with cold water. Determine both potassium and sodium in the filtrate first as mixed chlorids in the ordinary manner and then determine the potassium by means of platinum chlorid.

A rapid but only approximate determination of the alkali in soap is made in the following manner: Weigh a small quantity of the soap, treat with concentrated sulphuric acid, burn, repeat treatment with sulphuric acid, and burn again. Add a small amount of ammonium carbonate to the dish, cover, and heat. Repeat this a number of times till all bisulphates have changed to sulphates. Test the residue qualitatively to determine whether it is sodium or potassium sulphate, and calculate the residue to soda or potash, as the case may be.

## 7. Soda Lye.

(a) **CARBONATE AND HYDROXID**.<sup>c</sup> METHOD I (PRECIPITATION).—PROVISIONAL.

(1) **SOLUTIONS REQUIRED.**

A half-normal solution of hydrochloric acid; methyl orange and phenolphthalein indicators.

(2) **DETERMINATION.**

Weigh a large quantity of the sample in a weighing bottle, dissolve in carbon-dioxid-free water, and make up to a definite volume. Analyze aliquots of this solution. Titrate one portion with half-normal acid, using methyl orange as indicator, and note the total alkalinity thus found. Transfer another aliquot of the same size to a measuring flask and add enough barium chlorid to precipitate all carbonate, avoiding any unnecessary excess. Make the volume up to the mark with carbon-dioxid-free water, stopper, shake, and set aside to allow

<sup>a</sup> Benedikt and Lewkowitsch, *Oils, Fats, and Waxes*, p. 632.

<sup>b</sup> Benedikt and Lewkowitsch, *Oils, Fats, and Waxes*, page 630; U. S. Dept. Agr., Bureau of Chemistry, Circular No. 10, Revised, page 7.

<sup>c</sup> Sutton's *Volumetric Analysis*, 9th ed., page 56.

the precipitate to settle. When the liquid becomes clear, draw off one-half by means of a pipette and titrate with half-normal hydrochloric acid, using phenolphthalein as indicator. This number of cubic centimeters of half-normal acid multiplied by 2 gives the number of cubic centimeters of half-normal acid corresponding to the original amount taken. The last figure obtained represents sodium hydroxid and the difference between the first and last figures represents the sodium carbonate.

(b) CARBONATE AND HYDROXID,<sup>a</sup> METHOD II (CAMERON).—PROVISIONAL.

(1) SOLUTIONS REQUIRED.

A fifth-normal solution of potassium acid sulphate; methyl orange and phenolphthalein indicators.

(2) DETERMINATION.

Dilute with carbon-dioxid-free water an aliquot of the solution as prepared in Method I and add a few drops of phenolphthalein. Add a fifth-normal solution of potassium acid sulphate at the rate of about 1 drop per second, with constant stirring, until the pink color fades out and the solution becomes colorless. The reading thus obtained ( $n$ ) represents the sodium hydroxid and one-half of the sodium carbonate present, since the sodium carbonate is changed to sodium bicarbonate. Add methyl orange and continue the titration to the appearance of a pink color. This reading ( $m$ ) represents the sodium bicarbonate present, or one-half of the sodium carbonate;  $2m$  represents all the sodium carbonate present, and  $n-m$  the sodium hydroxid.

8. Tobacco and Tobacco Extract.

(a) NICOTIN, KISSLING METHOD.—OFFICIAL.

(1) SOLUTIONS REQUIRED.

(a) *Alcoholic soda*.—Dissolve 6 grams of sodium hydroxid in 40 cc of water and 60 cc of 90 per cent alcohol.

(b) *Sodium hydroxid*.—Dissolve 4 grams of sodium hydroxid in 1,000 cc of water.

(c) *Sulphuric acid*.—A standard solution.

(2) DETERMINATION.

Weigh from 5 to 6 grams of tobacco extract or 20 grams of finely powdered tobacco, which has been previously dried at 60° C. so as to allow it to be powdered, into a small beaker. Add 10 cc of the alcohol-soda solution and follow, in the case of the tobacco extract, with enough chemically pure powdered calcium carbonate to form a moist but not lumpy mass. Mix the whole thoroughly. Transfer this to a Soxhlet extractor and exhaust for about five hours with ether. Evaporate the ether at a low temperature by holding over the steam bath, and take up the residue with 50 cc of the dilute sodium hydroxid solution. Transfer this residue by means of water to a Kjeldahl flask, capable of holding about 500 cc, and distil in a current of steam, using a condenser through which water is flowing rapidly. Use a three-bend outflow tube, a few pieces of pumice, and a small piece of paraffin, to prevent bumping and frothing. Continue the distillation till all the nicotin has passed over, the distillate usually

<sup>a</sup> U. S. Dept. Agr., Bureau of Soils, Bul. 18, p. 77; by action of the association in 1907 this method was dropped.



varying from 400 to 500 cc. When the distillation is complete, only about 15 cc of the liquid should remain in the distillation flask. Titrate the distillate with standard sulphuric acid, using phenacetolin or cochineal as indicator. One molecule of sulphuric acid is equivalent to two molecules of nicotin.

### 9. Formaldehyde Solutions (Formalin, etc.).

#### (a) FORMALDEHYDE,<sup>a</sup> METHOD I (MODIFIED HYDROGEN PEROXID).—PROVISIONAL.

##### (1) SOLUTIONS REQUIRED.

A normal solution of sulphuric acid, a normal solution of sodium hydroxid, and a solution of purified litmus.

##### (2) DETERMINATION.

Measure out 50 cc of normal sodium hydroxid in a 500 cc Erlenmeyer flask and add 50 cc of hydrogen dioxid. Then add 3 cc of the formaldehyde solution under examination (the specific gravity of which has been previously determined), allowing the point of the pipette to almost reach the liquid in the flask. Place a funnel in the neck of the flask and put on the steam bath for five minutes, shaking occasionally during this time. Remove from the steam bath, wash the funnel with distilled water, cool the flask to about room temperature, and titrate the excess of sodium hydroxid with normal acid, using litmus as indicator. This cooling of the flask before titration with acid is necessary in order to get a sharp end reading with the litmus. From the volume of formaldehyde used and the specific gravity determine the per cent of formaldehyde.

#### (b) FORMALDEHYDE,<sup>b</sup> METHOD II (CYANID).—PROVISIONAL.

(To be used especially in solutions containing a small amount of formaldehyde.)

##### (1) SOLUTIONS REQUIRED.

(a) *Silver nitrate*.—A tenth-normal solution.

(b) *Ammonium sulpho-cyanate*.—A tenth-normal solution.

(c) *Potassium cyanid*.—A solution containing 3.1 grams to 500 cc of water.

(d) *Nitric acid*.—A 50 per cent solution.

##### (2) DETERMINATION.

Treat 15 cc of the silver nitrate with 6 drops of 50 per cent nitric acid in a 50 cc flask; add 10 cc of the solution of potassium cyanid and shake well. Then make the solution to the mark and titrate an aliquot of the filtrate (say 25 cc) according to the method of Volhard (page 23) with the tenth-normal solution of ammonium sulpho-cyanate for the excess of silver. Acidify another 15 cc portion of tenth-normal silver nitrate with 6 drops of 50 per cent nitric acid and treat with 10 cc of the potassium cyanid solution to which has been added a weighed quantity of the dilute formaldehyde solution. Make up the whole to 50 cc and titrate a 25 cc filtrate from it with tenth-normal ammonium sulpho-cyanate for the excess of silver as before. The difference between these results multiplied by 2 gives the amount of potassium cyanid that has been used by the formaldehyde in terms of tenth-normal ammonium sulpho-cyanate. Each cubic centimeter of this is equivalent to 3 milligrams of formaldehyde.

<sup>a</sup> Ber. d. chem. Ges., 1898, 31: 2979; J. Amer. Chem. Soc., 1905, 27: 1183.

<sup>b</sup> Zts. anal. Chem., 1897, 36: 18.

**10. Lime-Sulphur Dips and Lime-Sulphur-Salt Mixture.****(a) TOTAL SULPHUR,<sup>a</sup> AVERY METHOD.—PROVISIONAL.****(1) SOLUTIONS REQUIRED.**

(a) *Alkali solution*.—Use a saturated potassium hydroxid solution or a solution of sodium hydroxid containing 100 grams to 100 cc of water.

(b) *Barium chlorid*.—A 10 per cent solution.

(c) *Hydrogen peroxid*.—An approximately 3 per cent solution, free from sulphates. If the solution contains sulphates add freshly precipitated barium carbonate and shake occasionally for several hours, then filter and use the clear solution.

**(2) DETERMINATION.**

Measure 10 cc of the clear sample in a 100 cc measuring flask and fill to the mark. Analyze 10 cc aliquots of this solution. Treat with 3 cc of the caustic potash or soda solution, following by 50 cc of hydrogen peroxid free from sulphates. Heat on the steam bath for one-half hour exactly and then acidify with hydrochloric acid, precipitate with barium chlorid in the usual way in boiling solution, and finally weigh as barium sulphate.

**11. Lead Arsenate.**

See Appendix, page 239, for provisional methods adopted by the association in 1907.

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<sup>a</sup> U. S. Dept. Agr., Bureau of Chemistry, Bul. 90, p. 105.

## V. METHODS FOR THE ANALYSIS OF TANNING MATERIALS.—PROVISIONAL.

### 1. Crude Materials.

#### (a) MOISTURE IN SAMPLE AS RECEIVED.

Grind promptly and dry 10 grams as directed under "4. Evaporation and drying."

#### (b) PREPARATION FOR EXTRACTION.

• Dry the sample not above 60° C. and grind to pass through a 20-mesh sieve.

#### (c) MOISTURE IN PREPARED SAMPLE.

Dry 10 grams as directed under "4. Evaporation and drying," and calculate all subsequent determinations on the moisture-free basis.

#### (d) EXTRACTION.

Extract such a quantity of material as will give between 0.35 and 0.45 gram of tannin per 100 cc of solution in an extractor which permits the removal of the extract solution without allowing it to boil, using a layer of cotton to prevent fine material from passing over. Collect from 400 to 500 cc in this way, remove and continue extraction with a fresh portion of water at steam heat in the usual way until the material is free of tannin, testing the last few cubic centimeters of extract with gelatin-salt solution.

For spent materials approximate the above proportions as closely as practicable.

#### (e) ANALYSIS.

Heat the extract to 80° C. and proceed as directed under extracts. In case weaker solutions than the method specifies are employed the amount of hide powder used must be reduced in proportion to the quantity of tannin present.

### 2. Extracts.

#### (a) PREPARATION OF SOLUTION.

Grind solid extracts rapidly, and thoroughly mix. Heat fluid extracts to 50° C., thoroughly mix, cool to room temperature, and weigh from weighing

bottles. Dissolve in 900 cc of water at 80° C. such a quantity of the extract as will give from 0.35 to 0.45 gram of tannin in 100 cc of solution. Allow to cool slowly for from 12 to 20 hours at a temperature not below 20° C. and dilute to 1 liter.

(b) TOTAL SOLIDS.

Thoroughly mix the solution, immediately pipette 100 cc into a tared dish, evaporate, and dry as directed under "4. Evaporation and drying."

(c) SOLUBLE SOLIDS.

Add 75 cc of solution (kept at from 20° to 25° C. during filtration) to 2 grams of kaolin (free from soluble salts), stir, let stand 15 minutes, decant, and discard as much as possible of the supernatant liquid, and again add 75 cc of the tannin solution to the kaolin. Stir, and pour immediately on a 15 cm, No. 590 folded filter. Keep the filter full and the funnel and receiving vessel covered. Reject the first 150 cc of filtrate, evaporate and dry the next 100 cc (which must be as clear as practicable), as directed under "4. Evaporation and drying."

(d) NONTANNINS.

Prepare a sufficient quantity of hide powder in the following manner: Digest with 25 times its weight of water until thoroughly soaked; add 3 per cent of chrome alum in solution, agitate occasionally for several hours, and allow to stand over night. Wash, by squeezing through linen, until the wash water gives no precipitate with barium chlorid. Squeeze the hide, using a press if necessary, so that it contains from 70 to 75 per cent of water, and determine moisture (20 grams is a convenient quantity).

Add to 200 cc of the tannin solution such a quantity of the wet hide as contains from 12 to 13 grams of dry hide, shake for 10 minutes in a shaker and squeeze immediately through linen. Add 2 grams of kaolin to the filtrate, stir, and filter through a folded 20 cm filter (No. 1 F Swedish recommended), returning until clear. Evaporate and dry 100 cc as directed under "4. Evaporation and drying." Correct the weight of the residue for dilution caused by the water contained in the wet hide powder.

The nontannin filtrate must not give a precipitate with a gelatin-salt solution (1 per cent of gelatin and 10 per cent of salt).

(e) TANNIN.

The difference between the weight of the soluble solids and the corrected nontannin residue is the weight of tannin in 100 cc of solution.

### 3. Liquors.

(a) PREPARATION OF SOLUTION.

Dilute to contain approximately 0.7 gram of solids in 100 cc of solution.

(b) TOTAL SOLIDS.

Proceed as in extracts, 2 (b).

(c) SOLUBLE SOLIDS.

Proceed as in extracts, 2 (c).

## (d) NONTANNINS.

Shake 200 cc of solution as directed under extracts with such an amount of the wet chromed hide powder (2 (d)) as will give the following proportions:

Dry hide.	Tannin in 200 cc.
<i>Grams.</i>	<i>Gram.</i>
8 to 10.....	0.7 to 0.8
5 to 8.....	0.5 to 0.7
2 to 5.....	0.3 to 0.5
0 to 2.....	0 to 0.3

Evaporate and dry 100 cc as directed under "4. Evaporation and drying."

**4. Evaporation and Drying.**

Evaporate and dry for 16 hours in a combined evaporator and dryer at from 98° to 100° C.; or, after evaporating, dry for 12 hours on the bottom shelf of a water oven at from 98° to 100° C. Conduct evaporation and drying in flat-bottom glass dishes from 2½ to 3 inches in diameter.

**5. Acidity of Liquors.**

Dilute 100 cc of liquor to 500 cc. Add 2 grams of purified animal charcoal to 100 cc of the dilute liquor in a flask provided with a tube condenser. Heat to boiling with frequent shaking, cool, filter, and titrate an aliquot with tenth-normal alkali.

## **VI. GENERAL METHODS FOR THE ANALYSIS OF FOODS AND FEEDING STUFFS.**

### **1. Moisture.—Official.**

Dry a convenient quantity of the substance, representing about 2 grams of dry material, at the temperature of boiling water until it ceases to lose weight (approximately five hours), in a current of dry hydrogen or in vacuo. If the substance be held in a glass vessel the latter should not be in contact with the boiling water.

### **2. Ash.—Official.**

Char a convenient quantity of the substance, representing about 2 grams of the dry material, and burn until free of carbon at the lowest possible heat. If a carbon-free ash can not be obtained in this manner, exhaust the charred mass with water, collect the insoluble residue on a filter, burn till the ash is white or nearly so, and then add the filtrate to the ash and evaporate to dryness. Heat the whole to a low redness and weigh.

### **3. Crude Protein.—Official.**

Determine nitrogen as directed under fertilizers "4. (a) or (b)," page 5, and multiply the results by 6.25.

### **4. Albuminoid Nitrogen.—Official.**

#### **(a) PREPARATION OF STUTZER'S REAGENT.**

Prepare cupric hydroxid as follows: Dissolve 100 grams of pure copper sulphate in 5 liters of water, add 2.5 cc of glycerol, and then a dilute solution of sodium hydroxid until the liquid is just alkaline; filter, rub the precipitate up with water containing 5 cc of glycerol per liter, and wash by decantation or filtration until the washings are no longer alkaline. Rub the precipitate up again in a mortar with water containing 10 per cent of glycerol, thus preparing a uniform gelatinous mass that can be measured with a pipette. Determine the quantity of copper hydroxid per cubic centimeter of this mixture.

#### **(b) DETERMINATION.**

Place 0.7 gram of the substance in a beaker, add 100 cc of water, and heat to boiling, or, in case of substances rich in starch, heat on the water bath ten minutes; add a quantity of copper hydroxid mixture containing about 0.5 gram of the hydroxid; stir thoroughly, filter when cold, wash with cold water, and, without removing the precipitate from the filter, determine nitrogen according to one of the methods given for the determination of nitrogen in fertilizers (4. (a) or (b), p. 5), adding sufficient potassium sulphid solution to completely precipitate all copper and mercury. The filter papers used must be practically

free from nitrogen. If the samples consist of material rich in alkaline phosphates (such as seeds, seed residue, and oil cake), add from 1 to 2 cc of a concentrated solution of potassium or sodium alum, free from ammonia, just before adding the copper hydroxid, and mix well by stirring. This serves to decompose the alkaline phosphates. If this be not done copper phosphate and free alkali may be formed, and the protein-copper precipitate may be partially dissolved in the alkaline liquid.

#### 5. Crude Fat or Ether Extract.—Official.

##### (a) PREPARATION OF ANHYDROUS ETHER.

Wash any of the commercial brands of ether with two or three successive portions of distilled water, add solid sodium or potassium hydroxid, and let stand until most of the water has been abstracted from the ether. Decant into a dry bottle, add carefully cleaned metallic sodium cut into small pieces, and let stand until there is no further evolution of hydrogen gas. The ether thus dehydrated must be kept over metallic sodium, and should be lightly stoppered in order to allow any accumulating hydrogen gas to escape. It may be drawn off with a pipette as required.

##### (b) DETERMINATION.

###### (1) DIRECT METHOD.

Extract a convenient quantity of the substance, representing about 2 grams of the dry material, dried as for the determination of moisture, with anhydrous alcohol-free ether for sixteen hours. Dry the extract at the temperature of boiling water for one-half hour, remove from the oven to a desiccator, cool and weigh; continue this alternate drying and weighing at half-hour intervals until a minimum weight of fat is obtained. For most feeds a period of from one to one and one-half hours is required to obtain a minimum weight.

###### (2) INDIRECT METHOD.

Determine moisture as above, extract the dried substance for sixteen hours as directed under the direct method, dry again and regard the loss of weight as ether extract.

#### 6. Sucrose.

##### OPTICAL METHODS.

###### (a) GENERAL DIRECTIONS FOR RAW SUGARS,<sup>a</sup>—PROVISIONAL.

1. In general, make all sugar tests at 20° C.

2. Graduate the saccharimeter at 20° C. Dissolve 26.000 grams of pure sugar in water, and make the volume up to 100 metric cc (or 26.048 grams of pure sugar in 100 Mohr cc), all weighings to be made in air with brass weights. Complete the volume and make the polarization at 20° C. on an instrument graduated at 20° C. This should give an indication of 100 on the scale of the saccharimeter. For laboratories in which temperatures are usually higher than 20° C., it is permissible to graduate saccharimeters at any suitable temperature under the conditions specified above, providing that the analysis of the sugar be made at the same temperature—that is, that the volume be completed and the polarizations made at the temperature specified.

<sup>a</sup> International commission for unifying methods of sugar analysis, Zts. Rüb.-Zuck. Ind., 1900, 37: 357.

3. To prepare pure sugar, further purify the purest commercial sugar in the following manner: Prepare a hot saturated aqueous solution, precipitate the sugar with absolute ethyl alcohol, spin the sugar carefully in a small centrifugal machine, and wash in the latter with absolute alcohol. Redissolve the sugar thus obtained in water, again precipitate the saturated solution with alcohol, and wash as above. Dry the second crop of crystals between blotting paper and preserve in glass vessels for use. Determine the moisture still contained in the sugar and take this into account when weighing the sugar which is to be used.

(NOTE.—The Bureau of Chemistry is prepared to furnish pure sucrose for those who may need it for control work. Wherever this arrangement is not feasible quartz plates, the values of which have been determined by means of chemically pure sugar, shall serve for the control of the saccharimeters. This control of quartz plates by means of chemically pure sugar should, as a rule, apply only to the Bureau of Chemistry, which is to test the correctness of saccharimeters; for those who execute commercial analyses, the repeated control of the instruments is to be accomplished, now as before, by quartz plates.)

4. In effecting the polarization of substances containing sugar employ only half-shade or triple field instruments.

5. During the observation keep the apparatus in a fixed position and so far removed from the source of light that the polarization nicol is not warmed.

6. Sources of light may be gas, triple burner with metallic cylinder, lens, and reflector; gas lamps with Auer (Welsbach) burner; electric lamp; petroleum duplex lamp; sodium light. Make several readings and take the mean thereof, but no one reading may be neglected.

7. In making a polarization use the whole normal weight for 100 cc, or a multiple thereof, for any corresponding volume.

8. As clarifying and decolorizing agents use either subacetate of lead, alumina cream, or concentrated solution of alum. Boneblack and decolorizing powders are to be excluded.

9. After bringing the solution exactly to the mark at the proper temperature, and after wiping out the neck of the flask with filter paper, pour all of the well-shaken clarified sugar solution on a rapidly acting filter. Reject the first portions of the filtrate and use the rest, which must be perfectly clear for polarization.

#### (b) PREPARATION OF REAGENTS.—PROVISIONAL.

(1) *Lead subacetate solution*.—Prepare by boiling 430 grams of normal lead acetate, 130 grams of litharge, and 1,000 cc of water for half an hour. Allow the mixture to cool and settle and dilute the supernatant liquid to 1.25 specific gravity with recently boiled water. Solid lead subacetate may be substituted for the normal salt and litharge in the preparation of the solution.

(2) *Alumina cream*.—Prepare a cold saturated solution of alum in water and divide into two unequal portions. Add a slight excess of ammonium hydroxid to the larger portion and then add by degrees the remaining alum solution until a faintly acid reaction is secured.

#### (c) DETERMINATION OF SUCROSE IN THE ABSENCE OF RAFFINOSE.—OFFICIAL.<sup>a</sup>

Dissolve the normal weight of the substance in water, clarify with lead subacetate, and dilute to 100 cc. Filter and polarize the filtrate at 20° C. in a 200 mm tube. The reading obtained is the direct reading or polarization before

<sup>a</sup> In the presence of much levulose, as in honeys and fruit products, the optical method for sucrose gives too high results.



inversion. Free 50 cc of this filtrate from lead by treating with anhydrous sodium carbonate, sodium sulphate or potassium oxalate, place in a 100 cc flask, and add 25 cc of water. Then add, little by little, while rotating the flask, 5 cc of hydrochloric acid, containing 38.8 per cent of the acid. Heat the flask after mixing in a water bath which is at 70° C. The temperature of the solution in the flask should reach 67° to 69° C. in two and one-half to three minutes. Maintain a temperature of as nearly 69° C. as possible during seven to seven and one-half minutes, making a total time of heating of ten minutes. Remove the flask and cool the contents rapidly to 20° C. and dilute to 100 cc. Polarize this solution in a tube provided with a lateral branch and a water jacket, passing a current of water around the tube to maintain a temperature of 20° C.

The inversion may also be accomplished as follows: To 50 cc of the clarified solution, freed from lead, add 5 cc of a 38.8 per cent solution of hydrochloric acid and set aside during a period of twenty-four hours at a temperature not below 20° C.; or if the temperature be above 25° C. set aside for ten hours. Make up to 100 cc at 20° C. and polarize at that temperature. This reading must be multiplied by two, which gives the invert reading. In case it is necessary to work at a temperature other than 20° C., which is allowable within narrow limits, the volumes must be completed and both direct and invert polarizations must be made at exactly the same temperature. The sucrose is calculated by the following formula:

$$S = \frac{100(P - I)}{142.66 - \frac{T}{2}}$$

S = per cent of sucrose.

P = direct reading.

I = invert reading.

T = temperature at which readings are made.

#### (d) DETERMINATION OF SUCROSE AND RAFFINOSE.—OFFICIAL.

(Of value chiefly in the analysis of beet sugars.)

If the direct reading be more than 1 degree higher than the per cent of sucrose as calculated by the formula given above, raffinose is probably present, and sucrose and raffinose should be calculated by the following formula of Croydt:

P = the direct reading.

I = the invert reading.

S = the percentage of sucrose.

R = the percentage of anhydrous raffinose.

$$S = \frac{0.5188 P - I}{0.8454}, R = \frac{P - S}{1.85}$$

#### CHEMICAL METHODS.—PROVISIONAL.

##### (a) DETERMINATION OF SUCROSE BY INVERSION AND REDUCTION.

Determine the reducing sugars as invert sugar in the sample and calculate them by the proper table for invert sugar-sucrose mixtures (pages 43, 45), invert the solution by one of the methods given under (c), exactly neutralize the acid, determine the reducing sugars, and calculate them from the table for invert sugar alone, page 45. Deduct the percentage of invert sugar obtained before inversion from the amount obtained after inversion. This is the amount due

to sucrose. Multiply this by 0.95 to obtain the sucrose. The solution should be diluted in both determinations so that not more than 245 mg of invert sugar are present in the amount taken for the reduction, and should be properly clarified with normal lead acetate and have the excess of lead removed before the determinations.

### 7. Reducing Sugars.

(See also Appendix, p. 241, for methods and tables of Munson and Walker.)

#### (a) DETERMINATIONS REQUIRING THE USE OF SOXHLET'S MODIFICATION OF FEHLING'S SOLUTION.

##### (1) PREPARATION OF REAGENTS.—PROVISIONAL.

(a) *Copper sulphate solution*.—Dissolve 34.639 grams of  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$  in water and dilute to 500 cc.

(b) *Alkaline tartrate solution*.—Dissolve 173 grams of Rochelle salts and 50 grams of sodium hydroxid in water and dilute to 500 cc.

(c) *Mixed solution*.—Mix equal volumes of solutions (a) and (b) immediately before use.

##### (2) VOLUMETRIC METHODS.

#### (a) *Approximate method for rapid work*.—Provisional.

(Applicable to invert sugar and dextrose.)

Place 10 cc of the mixed copper reagent in a large test tube and add 10 cc of distilled water. Heat to boiling, and gradually add small portions of the solution of the material to be tested until the copper has been completely precipitated, boiling to complete the reaction after each addition. Two minutes' boiling is required for complete precipitation when the full amount of sugar solution has been added in one portion. When the end reaction is nearly reached and the amount of sugar solution to be added can no longer be judged by the color of the solution, remove a small portion of the liquid and filter rapidly into a small porcelain crucible or on a test plate; acidify with dilute acetic acid, and test for copper with a dilute solution of potassium ferrocyanid. The sugar solution should be of such strength as will give a burette reading of 15 to 20 cc, and the number of successive additions should be as small as possible.

Since the factor of calculation varies with the minute details of manipulation, every operator must determine a factor for himself, using a known solution of a pure sample of the sugar that he desires to determine, and keeping the conditions the same as those used for the determinations.

Standardize the solution for invert sugar in the following manner:

Dissolve 4.75 grams of pure sucrose in 75 cc of water, add 5 cc of 38.8 per cent hydrochloric acid, and invert as under the official method for sucrose, page 41. Neutralize the acid exactly with sodium hydroxid and dilute to 1 liter. Ten cubic centimeters of this solution contains 0.050 gram of invert sugar, which should reduce 10 cc of the copper solution; the copper solution should never be taken as a standard, but should be checked against the sugar. In case this method is used for determining dextrose, pure dextrose must be used in standardizing the solution.

#### (b) *Soxhlet's method*.—Provisional.

Make a preliminary titration to determine the approximate percentage of reducing sugar in the material under examination. Prepare a solution which contains approximately 1 per cent of reducing sugar. Place in a beaker 100 cc

of the mixed copper reagent and approximately the amount of the sugar solution for its complete reduction. Boil for two minutes. Filter through a folded filter and test a portion of the filtrate for copper by use of acetic acid and potassium ferrocyanid. Repeat the test, varying the volume of sugar solution, until two successive amounts are found which differ by 0.1 cc, one giving complete reduction and the other leaving a small amount of copper in solution. The mean of these two readings is taken as the volume of the solution required for the complete precipitation of 100 cc of the copper reagent.

Under these conditions 100 cc of the mixed copper reagent require 0.475 gram of anhydrous dextrose or 0.494 gram of invert sugar for complete reduction. Calculate the percentage by the following formula:

V=the volume of the sugar solution required for the complete reduction of 100 cc of the copper reagent.

W=the weight of the sample in 1 cc of the sugar solution.

$$\text{Then } \frac{100 \times 0.475}{VW} = \text{per cent of dextrose,}$$

$$\text{or } \frac{100 \times 0.494}{VW} = \text{per cent of invert sugar.}$$

### (3) GRAVIMETRIC METHODS.

#### (a) *Invert sugar.—Provisional.*

(a) Determination in materials containing 1 per cent or less of invert sugar and 99 per cent or more of sucrose:

Prepare the solution of the material to be examined so as to contain 20 grams in 100 cc, free from suspended impurities by filtration and from soluble impurities by normal lead acetate, removing the excess of lead by means of sodium carbonate. Place 50 cc of the mixed copper reagent and 50 cc of the sugar solution in a beaker of 250 cc capacity. Heat this mixture at such a rate that approximately four minutes are required to bring it to the boiling point, and boil for exactly two minutes. Add 100 cc of cold, recently boiled, distilled water. Filter immediately through asbestos and determine the copper by one of the methods given under (c), page 51.

Obtain the corresponding percentage of invert sugar by the use of the following table:

*Hertzfeld's table for the determination of invert sugar in materials containing 1 per cent or less of invert sugar and 99 per cent or more of sucrose.*

Copper reduced by 10 grams of material.	Invert sugar.	Copper reduced by 10 grams of material.	Invert sugar.	Copper reduced by 10 grams of material.	Invert sugar.
<i>Milligrams.</i>	<i>Per cent.</i>	<i>Milligrams.</i>	<i>Per cent.</i>	<i>Milligrams.</i>	<i>Per cent.</i>
50	0.05	120	0.40	190	0.79
55	0.07	125	0.43	195	0.82
60	0.09	130	0.45	200	0.85
65	0.11	135	0.48	205	0.88
70	0.14	140	0.51	210	0.90
75	0.16	145	0.53	215	0.93
80	0.19	150	0.56	220	0.96
85	0.21	155	0.59	225	0.99
90	0.24	160	0.62	230	1.02
95	0.27	165	0.65	235	1.05
100	0.30	170	0.68	240	1.07
105	0.32	175	0.71	245	1.10
110	0.35	180	0.74		
115	0.38	185	0.76		

(b) Determination in materials in which of the total sugars present, 1 per cent or more is invert sugar and 99 per cent or less is sucrose:

Prepare a solution of the material to be examined in such a manner that it contains 20 grams in 100 cc after clarification and removal of the excess of lead. Prepare a series of solutions in large test tubes by adding 1, 2, 3, 4, and 5 cc of this solution to each tube successively. Add 5 cc of the mixed copper reagent to each, heat to boiling, boil two minutes, and filter. Note the volume of sugar solution which gives the filtrate lightest in tint, but still distinctly blue. Place twenty times this volume of the sugar solution in a 100 cc flask, dilute to the mark, and mix well. Use 50 cc of the solution for the determination, which is conducted as described under (a). For the calculation of the result use the following formulas and table of factors of Meissl and Hiller:

Let Cu=the weight of copper obtained;

P=the polarization of the sample;

W=the weight of the sample in the 50 cc of the solution used for determination;

F=the factor obtained from the table for conversion of copper to invert sugar;

$$\frac{\text{Cu}}{2} = \text{approximate absolute weight of invert sugar} = Z;$$

$$Z \times \frac{100}{W} = \text{approximate per cent of invert sugar} = y;$$

$$\frac{100 P}{P+y} = R, \text{ relative number for sucrose};$$

$$100 - R = I, \text{ relative number for invert sugar};$$

$$\frac{\text{Cu } F}{W} = \text{per cent of invert sugar}.$$

Z facilitates reading the vertical columns; and the ratio of R to I, the horizontal columns of the table, for the purpose of finding the factor (F) for calculation of copper to invert sugar.

Example.—The polarization of a sugar is 86.4, and 3.256 grams of it (W) are equivalent to 0.290 gram of copper. Then:

$$\frac{\text{Cu}}{2} = \frac{0.290}{2} = 0.145 = Z$$

$$Z \times \frac{100}{W} = 0.145 \times \frac{100}{3.256} = 4.45 = Y$$

$$\frac{100 P}{P+y} = \frac{86.4}{86.4+4.45} = 95.1 = R$$

$$100 - R = 100 - 95.1 = I = 4.9$$

$$R : I = 95.1 : 4.9$$

By consulting the table it will be seen that the vertical column headed 150 is nearest to Z, 145, and the horizontal column headed 95:5 is nearest to the ratio of R to I, 95.1:4.9. Where these columns meet, we find the factor 51.2 which enters into the final calculation:

$$\frac{\text{Cu } F}{W} = \frac{0.290 \times 51.2}{3.256} = 4.56 \text{ per cent of invert sugar}.$$

*Meissl and Hiller's factors for determinations in materials in which, of the total sugars present, 1 per cent or more is invert sugar, and 99 per cent or less is sucrose.*

Ratio of sucrose to invert sugar = R : I.	Approximate absolute weight of invert sugar = Z.						
	200 milli-grams.	175 milli-grams.	150 milli-grams.	125 milli-grams.	100 milli-grams.	75 milli-grams.	50 milli-grams.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
0:100	56.4	55.4	54.5	53.8	53.2	53.0	53.0
10:90	56.3	55.3	54.4	53.8	53.2	52.9	52.9
20:80	56.2	55.2	54.3	53.7	53.2	52.7	52.7
30:70	56.1	55.1	54.2	53.7	53.2	52.6	52.6
40:60	55.9	55.0	54.1	53.6	53.1	52.5	52.4
50:50	55.7	54.9	54.0	53.5	53.1	52.3	52.2
60:40	55.6	54.7	53.8	53.2	52.8	52.1	51.9
70:30	55.5	54.5	53.5	52.9	52.5	51.9	51.6
80:20	55.4	54.3	53.3	52.7	52.2	51.7	51.3
90:10	54.6	53.6	53.1	52.6	52.1	51.6	51.2
91:9	54.1	53.6	52.6	52.1	51.6	51.2	50.7
92:8	53.6	53.1	52.1	51.6	51.2	50.7	50.3
93:7	53.6	53.1	52.1	51.2	50.7	50.3	49.8
94:6	53.1	52.6	51.6	50.7	50.3	49.8	48.9
95:5	52.6	52.1	51.2	50.3	49.4	48.9	48.5
96:4	52.1	51.2	50.7	49.8	48.9	47.7	46.9
97:3	50.7	50.3	49.8	48.9	47.7	46.2	45.1
98:2	49.9	48.9	48.5	47.3	45.8	43.3	40.0
99:1	47.7	47.3	46.5	45.1	43.3	41.2	38.1

In case there is no sucrose present, the following table may be used instead of the factors given in the preceding table.

*Meissl table for the determination of invert sugar alone.*

[According to Wein.]

Copper.	Invert sugar.	Copper.	Invert sugar.	Copper.	Invert sugar.	Copper.	Invert sugar.
<i>Mg.</i>	<i>Mg.</i>	<i>Mg.</i>	<i>Mg.</i>	<i>Mg.</i>	<i>Mg.</i>	<i>Mg.</i>	<i>Mg.</i>
90	46.9	125	65.5	160	84.3	195	103.4
91	47.4	126	66.0	161	84.8	196	104.0
92	47.9	127	66.5	162	85.4	197	104.6
93	48.4	128	67.1	163	85.9	198	105.1
94	48.9	129	67.6	164	86.5	199	105.7
95	49.5	130	68.1	165	87.0	200	106.3
96	50.0	131	68.7	166	87.6	201	106.8
97	50.5	132	69.2	167	88.1	202	107.4
98	51.1	133	69.7	168	88.6	203	107.9
99	51.6	134	70.3	169	89.2	204	108.5
100	52.1	135	70.8	170	89.7	205	109.1
101	52.7	136	71.3	171	90.3	206	109.6
102	53.2	137	71.9	172	90.8	207	110.2
103	53.7	138	72.4	173	91.4	208	110.8
104	54.3	139	72.9	174	91.9	209	111.3
105	54.8	140	73.5	175	92.4	210	111.9
106	55.3	141	74.0	176	93.0	211	112.5
107	55.9	142	74.5	177	93.5	212	113.0
108	56.4	143	75.1	178	94.1	213	113.6
109	56.9	144	75.6	179	94.6	214	114.2
110	57.5	145	76.1	180	95.2	215	114.7
111	58.0	146	76.7	181	95.7	216	115.3
112	58.5	147	77.2	182	96.2	217	115.8
113	59.1	148	77.8	183	96.8	218	116.4
114	59.6	149	78.3	184	97.3	219	117.0
115	60.1	150	78.9	185	97.8	220	117.5
116	60.7	151	79.4	186	98.4	221	118.1
117	61.2	152	80.0	187	99.0	222	118.7
118	61.7	153	80.5	188	99.5	223	119.2
119	62.3	154	81.0	189	100.1	224	119.8
120	62.8	155	81.6	190	100.6	225	120.4
121	63.3	156	82.1	191	101.2	226	120.9
122	63.9	157	82.7	192	101.7	227	121.5
123	64.4	158	83.2	193	102.3	228	122.1
124	64.9	159	83.8	194	102.9	229	122.6

*Meissl table for the determination of invert sugar alone—Continued.*

Copper.	Invert sugar.	Copper.	Invert sugar.	Copper.	Invert sugar.	Copper	Invert sugar.
<i>Mg.</i>	<i>Mg.</i>	<i>Mg.</i>	<i>Mg.</i>	<i>Mg.</i>	<i>Mg.</i>	<i>Mg.</i>	<i>Mg.</i>
230	123.2	280	151.9	330	181.6	380	212.4
231	123.8	281	152.5	331	182.2	381	213.0
232	124.3	282	153.1	332	182.8	382	213.6
233	124.9	283	153.7	333	183.5	383	214.3
234	125.5	284	154.3	334	184.1	384	214.9
235	126.0	285	154.9	335	184.7	385	215.5
236	126.6	286	155.5	336	185.4	386	216.1
237	127.2	287	156.1	337	186.0	387	216.8
238	127.8	288	156.7	338	186.6	388	217.4
239	128.3	289	157.2	339	187.2	389	218.0
240	128.9	290	157.8	340	187.8	390	218.7
241	129.5	291	158.4	341	188.4	391	219.3
242	130.0	292	159.0	342	189.0	392	219.9
243	130.6	293	159.6	343	189.6	393	220.5
244	131.2	294	160.2	344	190.2	394	221.2
245	131.8	295	160.8	345	190.8	395	221.8
246	132.3	296	161.4	346	191.4	396	222.4
247	132.9	297	162.0	347	192.0	397	223.1
248	133.5	298	162.6	348	192.6	398	223.7
249	134.1	299	163.2	349	193.2	399	224.3
250	134.6	300	163.8	350	193.8	400	224.9
251	135.2	301	164.4	351	194.4	401	225.7
252	135.8	302	165.0	352	195.0	402	226.4
253	136.3	303	165.6	353	195.6	403	227.1
254	136.9	304	166.2	354	196.2	404	227.8
255	137.5	305	166.8	355	196.8	405	228.6
256	138.1	306	167.3	356	197.4	406	229.3
257	138.6	307	167.9	357	198.0	407	230.0
258	139.2	308	168.5	358	198.6	408	230.7
259	139.8	309	169.1	359	199.2	409	231.4
260	140.4	310	169.7	360	199.8	410	232.1
261	140.9	311	170.3	361	200.4	411	232.8
262	141.5	312	170.9	362	201.1	412	233.5
263	142.1	313	171.5	363	201.7	413	234.3
264	142.7	314	172.1	364	202.3	414	235.0
265	143.2	315	172.7	365	203.0	415	235.7
266	143.8	316	173.3	366	203.6	416	236.4
267	144.4	317	173.9	367	204.2	417	237.1
268	144.9	318	174.5	368	204.8	418	237.8
269	145.5	319	175.1	369	205.5	419	238.5
270	146.1	320	175.6	370	206.1	420	239.2
271	146.7	321	176.2	371	206.7	421	239.9
272	147.2	322	176.8	372	207.3	422	240.6
273	147.8	323	177.4	373	208.0	423	241.3
274	148.4	324	178.0	374	208.6	424	242.0
275	149.0	325	178.6	375	209.2	425	242.7
276	149.5	326	179.2	376	209.9	426	243.4
277	150.1	327	179.8	377	210.5	427	244.1
278	150.7	328	180.4	378	211.1	428	244.9
279	151.3	329	181.0	379	211.7	429	245.6
						430	246.3

*(b) Maltose.—Provisional.*

Place 50 cc of the mixed copper reagent in a beaker and heat to the boiling point. While boiling briskly add 25 cc of the maltose solution containing not more than 0.250 gram of maltose and boil for four minutes. Filter immediately through asbestos and determine the amount of copper reduced by one of the

methods given under (c), page 51. Obtain the weight of maltose equivalent to the weight of copper found from the following table:

*Table for the determination of maltose.*

[According to Wein.]

Milli-grams of cop-per.	Milli-grams of cu-prous oxid.	Milli-grams of mal-tose.	Milli-grams of cop-per.	Milli-grams of cu-prous oxid.	Milli-grams of mal-tose.	Milli-grams of cop-per.	Milli-grams of cu-prous oxid.	Milli-grams of mal-tose.	Milli-grams of cop-per.	Milli-grams of cu-prous oxid.	Milli-grams of mal-tose.
31	34.9	26.1	86	96.8	74.1	141	158.7	123.3	196	220.7	172.5
32	36.0	27.0	87	97.9	75.0	142	159.9	124.2	197	221.8	173.4
33	37.2	27.9	88	99.1	75.9	143	161.0	125.1	198	222.9	174.3
34	38.3	28.7	89	100.2	76.8	144	162.1	126.0	199	224.0	175.2
35	39.4	29.6	90	101.3	77.7	145	163.2	126.9	200	225.2	176.1
36	40.5	30.5	91	102.4	78.6	146	164.4	127.8	201	226.3	177.0
37	41.7	31.3	92	103.6	79.5	147	165.5	128.7	202	227.4	177.9
38	42.8	32.2	93	104.7	80.3	148	166.6	129.6	203	228.5	178.7
39	43.9	33.1	94	105.8	81.2	149	167.7	130.5	204	229.7	179.6
40	45.0	33.9	95	107.0	82.1	150	168.9	131.4	205	230.8	180.5
41	46.2	34.8	96	108.1	83.0	151	170.0	132.3	206	231.9	181.4
42	47.3	35.7	97	109.2	83.9	152	171.1	133.2	207	233.0	182.3
43	48.4	36.5	98	110.3	84.8	153	172.3	134.1	208	234.2	183.2
44	49.5	37.4	99	111.5	85.7	154	173.4	135.0	209	235.3	184.1
45	50.7	38.3	100	112.6	86.6	155	174.5	135.9	210	236.4	185.0
46	51.8	39.1	101	113.7	87.5	156	175.6	136.8	211	237.6	185.9
47	52.9	40.0	102	114.8	88.4	157	176.8	137.7	212	238.7	186.8
48	54.0	40.9	103	116.0	89.2	158	177.9	138.6	213	239.8	187.7
49	55.2	41.8	104	117.1	90.1	159	179.0	139.5	214	240.9	188.6
50	56.3	42.6	105	118.2	91.0	160	180.1	140.4	215	242.1	189.5
51	57.4	43.5	106	119.3	91.9	161	181.3	141.3	216	243.2	190.4
52	58.5	44.4	107	120.5	92.8	162	182.4	142.2	217	244.3	191.2
53	59.7	45.2	108	121.6	93.7	163	183.5	143.1	218	245.4	192.1
54	60.8	46.1	109	122.7	94.6	164	184.6	144.0	219	246.6	193.0
55	61.9	47.0	110	123.8	95.5	165	185.8	144.9	220	247.7	193.9
56	63.0	47.8	111	125.0	96.4	166	186.9	145.8	221	248.7	194.8
57	64.2	48.7	112	126.1	97.3	167	188.0	146.7	222	249.9	195.7
58	65.3	49.6	113	127.2	98.1	168	189.1	147.6	223	251.0	196.6
59	66.4	50.4	114	128.3	99.0	169	190.3	148.5	224	252.4	197.5
60	67.6	51.3	115	129.6	99.9	170	191.4	149.4	225	253.3	198.4
61	68.7	52.2	116	130.6	100.8	171	192.5	150.3	226	254.4	199.3
62	69.8	53.1	117	131.7	101.7	172	193.6	151.2	227	255.6	200.2
63	70.9	53.9	118	132.8	102.6	173	194.8	152.0	228	256.7	201.1
64	72.1	54.8	119	134.0	103.5	174	195.9	152.9	229	257.8	202.0
65	73.2	55.7	120	135.1	104.4	175	197.0	153.8	230	258.9	202.9
66	74.3	56.6	121	136.2	105.3	176	198.1	154.7	231	260.1	203.8
67	75.4	57.4	122	137.4	106.2	177	199.3	155.6	232	261.2	204.7
68	76.6	58.3	123	138.5	107.1	178	200.4	156.5	233	262.3	205.6
69	77.7	59.2	124	139.6	108.0	179	201.5	157.4	234	263.4	206.5
70	78.8	60.1	125	140.7	108.9	180	202.6	158.3	235	264.6	207.4
71	79.9	61.0	126	141.9	109.8	181	203.8	159.2	236	265.7	208.3
72	81.1	61.8	127	143.0	110.7	182	204.9	160.1	237	266.8	209.1
73	82.2	62.7	128	144.1	111.6	183	206.0	160.9	238	268.0	210.0
74	83.3	63.6	129	145.2	112.5	184	207.1	161.8	239	269.1	210.9
75	84.4	64.5	130	146.4	113.4	185	208.3	162.7	240	270.2	211.8
76	85.6	65.4	131	147.5	114.3	186	209.4	163.6	241	271.3	212.7
77	86.7	66.2	132	148.6	115.2	187	210.5	164.5	242	272.5	213.6
78	87.8	67.1	133	149.7	116.1	188	211.7	165.4	243	273.6	214.5
79	88.9	68.0	134	150.9	117.0	189	212.8	166.3	244	274.7	215.4
80	90.1	68.9	135	152.0	117.9	190	213.9	167.2	245	275.8	216.3
81	91.2	69.7	136	153.1	118.8	191	215.0	168.1	246	277.0	217.2
82	92.3	70.6	137	154.2	119.7	192	216.2	169.0	247	278.1	218.1
83	93.4	71.5	138	155.4	120.6	193	217.3	169.8	248	279.2	219.0
84	94.6	72.4	139	156.5	121.5	194	218.4	170.7	249	280.3	219.9
85	95.7	73.2	140	157.6	122.4	195	219.5	171.6	250	281.5	220.8

Table for the determination of maltose—Continued.

Milli-grams of cop-per.	Milli-grams of cuprous oxid.	Milli-grams of mal-tose.	Milli-grams of cop-per.	Milli-grams of cuprous oxid.	Milli-grams of mal-tose.	Milli-grams of cop-per.	Milli-grams of cuprous oxid.	Milli-grams of mal-tose.	Milli-grams of cop-per.	Milli-grams of cuprous oxid.	Milli-grams of mal-tose.
251	282.6	221.7	264	297.2	233.4	277	311.9	245.1	290	326.5	256.6
252	283.7	222.6	265	298.3	234.3	278	313.0	246.0	291	327.4	257.5
253	284.8	223.5	266	299.5	235.2	279	314.1	246.9	292	328.7	258.4
254	286.0	224.4	267	300.6	236.1	280	315.2	247.8	293	329.9	259.3
255	287.1	225.3	268	301.7	237.0	281	316.4	248.7	294	331.0	260.2
256	288.2	226.2	269	302.8	237.9	282	317.5	249.6	295	332.1	261.1
257	289.3	227.1	270	304.0	238.8	283	318.6	250.4	296	333.2	262.0
258	290.5	228.0	271	305.1	239.7	284	319.7	251.3	297	334.4	262.8
259	291.6	228.9	272	306.2	240.6	285	320.9	252.2	298	335.5	263.7
260	292.7	229.8	273	307.3	241.5	286	322.0	253.1	299	336.6	264.6
261	293.8	230.7	274	308.5	242.4	287	323.1	254.0	300	337.8	265.5
262	295.0	231.6	275	309.6	243.3	288	324.2	254.9			
263	296.1	232.5	276	310.7	244.2	289	325.4	255.8			

## (c) Lactose.—Official.

Place 50 cc of the mixed copper reagent in a beaker and heat to the boiling point. While boiling briskly add 100 cc of the lactose solution containing not more than 0.300 gram of lactose and boil for six minutes. Filter immediately through asbestos and determine the amount of copper reduced by one of the methods given under (c), page 51. Obtain the weight of lactose equivalent to the weight of copper found from the following table:

Table for the determination of lactose (Sohxlet-Wein).

Milli-grams of cop-per.	Milli-grams of lac-tose.	Milli-grams of cop-per.	Milli-grams of lac-tose.	Milli-grams of cop-per.	Milli-grams of lac-tose.	Milli-grams of cop-per.	Milli-grams of lac-tose.	Milli-grams of cop-per.	Milli-grams of lac-tose.
100	71.6	125	90.1	150	108.8	175	127.8	200	146.9
101	72.4	126	90.9	151	109.6	176	128.5	201	147.7
102	73.1	127	91.6	152	110.3	177	129.3	202	148.5
103	73.8	128	92.4	153	111.1	178	130.1	203	149.2
104	74.6	129	93.1	154	111.9	179	130.8	204	150.0
105	75.3	130	93.8	155	112.6	180	131.6	205	150.7
106	76.1	131	94.6	156	113.4	181	132.4	206	151.5
107	76.8	132	95.3	157	114.1	182	133.1	207	152.2
108	77.6	133	96.1	158	114.9	183	133.9	208	153.0
109	78.3	134	96.9	159	115.6	184	134.7	209	153.7
110	79.0	135	97.6	160	116.4	185	135.4	210	154.5
111	79.8	136	98.3	161	117.1	186	136.2	211	155.2
112	80.5	137	99.1	162	117.9	187	137.0	212	156.0
113	81.3	138	99.8	163	118.6	188	137.7	213	156.7
114	82.0	139	100.5	164	119.4	189	138.5	214	157.5
115	82.7	140	101.3	165	120.2	190	139.3	215	158.2
116	83.5	141	102.0	166	120.9	191	140.0	216	159.0
117	84.2	142	102.8	167	121.7	192	140.8	217	159.7
118	85.0	143	103.5	168	122.4	193	141.6	218	160.4
119	85.7	144	104.3	169	123.2	194	142.3	219	161.2
120	86.4	145	105.1	170	123.9	195	143.1	220	161.9
121	87.2	146	105.8	171	124.7	196	143.9	221	162.7
122	87.9	147	106.6	172	125.5	197	144.6	222	163.4
123	88.7	148	107.3	173	126.2	198	145.4	223	164.2
124	89.4	149	108.1	174	127.0	199	146.2	224	164.9



Table for the determination of lactose (Saxhlet-Wein)—Continued.

Milli-grams of cop-per.	Milli-grams of lac-tose.	Milli-grams of cop-per.	Milli-grams of lac-tose.	Milli-grams of cop-per.	Milli-grams of lac-tose.	Milli-grams of cop-per.	Milli-grams of lac-tose.	Milli-grams of cop-per.	Milli-grams of lac-tose.
225	165.7	261	193.3	297	221.9	333	250.0	369	279.6
226	166.4	262	194.1	298	222.7	334	250.8	370	280.5
227	167.2	263	194.9	299	223.5	335	251.6	371	281.4
228	167.9	264	195.7	300	224.4	336	252.5	372	282.2
229	168.6	265	196.4	301	225.2	337	253.3	373	283.1
230	169.4	266	197.2	302	225.9	338	254.1	374	283.9
231	170.1	267	198.0	303	226.7	339	254.9	375	284.8
232	170.9	268	198.8	304	227.5	340	255.7	376	285.7
233	171.6	269	199.5	305	228.3	341	256.5	377	286.5
234	172.4	270	200.3	306	229.1	342	257.4	378	287.4
235	173.1	271	201.1	307	229.8	343	258.2	379	288.2
236	173.9	272	201.9	308	230.6	344	259.0	380	289.1
237	174.6	273	202.7	309	231.4	345	259.8	381	289.9
238	175.4	274	203.5	310	232.2	346	260.6	382	290.8
239	176.2	275	204.3	311	232.9	347	261.4	383	291.7
240	176.9	276	205.1	312	233.7	348	262.3	384	292.5
241	177.7	277	205.9	313	234.5	349	263.1	385	293.4
242	178.5	278	206.7	314	235.3	350	263.9	386	294.2
243	179.3	279	207.5	315	236.1	351	264.7	387	295.1
244	180.1	280	208.3	316	236.8	352	265.5	388	296.0
245	180.8	281	209.1	317	237.6	353	266.3	389	296.8
246	181.6	282	209.9	318	238.4	354	267.2	390	297.7
247	182.4	283	210.7	319	239.2	355	268.0	391	298.5
248	183.2	284	211.5	320	240.0	356	268.8	392	299.4
249	184.0	285	212.3	321	240.7	357	269.6	393	300.3
250	184.8	286	213.1	322	241.5	358	270.4	394	301.1
251	185.5	287	213.9	323	242.3	359	271.2	395	302.0
252	186.3	288	214.7	324	243.1	360	272.1	396	302.8
253	187.1	289	215.5	325	243.9	361	272.9	397	303.7
254	187.9	290	216.3	326	244.6	362	273.7	398	304.6
255	188.7	291	217.1	327	245.4	363	274.5	399	305.4
256	189.4	292	217.9	328	246.2	364	275.3	400	306.3
257	190.2	293	218.7	329	247.0	365	276.2		
258	191.0	294	219.5	330	247.7	366	277.1		
259	191.8	295	220.3	331	248.5	367	277.9		
260	192.5	296	221.1	332	249.2	368	278.8		

## (b) DETERMINATION REQUIRING THE USE OF ALLIHN'S MODIFICATION OF FEHLING'S SOLUTION.—PROVISIONAL.

## (1) PREPARATION OF REAGENTS.

(a) *Copper sulphate solution*.—Dissolve 34.639 grams of  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$  in water and dilute to 500 cc.

(b) *Alkaline tartrate solution*.—Dissolve 173 grams of Rochelle salts and 125 grams of potassium hydroxid in water and dilute to 500 cc.

## (2) GRAVIMETRIC METHOD FOR THE DETERMINATION OF DEXTROSE.

Place 30 cc of the copper solution, 30 cc of the alkaline tartrate solution, and 60 cc of water in a beaker and heat to boiling. Add 25 cc of the solution of the material to be examined, which must be so prepared as not to contain more than 0.250 gram of dextrose, and boil for two minutes. Filter immediately through asbestos without diluting, and obtain the weight of copper by

one of the methods given under (c), page 51. The corresponding weight of dextrose is found from the following table:

*Allihn's table for the determination of dextrose.*

Milli-grams of cop-per.	Milli-grams of cuprous oxid.	Milli-grams of dex-trose.	Milli-grams of cop-per.	Milli-grams of cuprous oxid.	Milli-grams of dex-trose.	Milli-grams of cop-per.	Milli-grams of cuprous oxid.	Milli-grams of dex-trose.	Milli-grams of cop-per.	Milli-grams of cuprous oxid.	Milli-grams of dex-trose.
11	12.4	6.6	71	79.9	36.3	131	147.5	66.7	191	215.0	97.8
12	13.5	7.1	72	81.1	36.8	132	148.6	67.2	192	216.2	98.4
13	14.6	7.6	73	82.2	37.3	133	149.7	67.7	193	217.3	98.9
14	15.8	8.1	74	83.3	37.8	134	150.9	68.2	194	218.4	99.4
15	16.9	8.6	75	84.4	38.3	135	152.0	68.8	195	219.5	100.0
16	18.0	9.0	76	85.6	38.8	136	153.1	69.3	196	220.7	100.5
17	19.1	9.5	77	86.7	39.3	137	154.2	69.8	197	221.8	101.0
18	20.3	10.0	78	87.8	39.8	138	155.4	70.3	198	222.9	101.5
19	21.4	10.5	79	88.9	40.3	139	156.5	70.8	199	224.0	102.0
20	22.5	11.0	80	90.1	40.8	140	157.6	71.3	200	225.2	102.6
21	23.6	11.5	81	91.2	41.3	141	158.7	71.8	201	226.3	103.1
22	24.8	12.0	82	92.3	41.8	142	159.9	72.3	202	227.4	103.7
23	25.9	12.5	83	93.4	42.3	143	161.0	72.9	203	228.5	104.2
24	27.0	13.0	84	94.6	42.8	144	162.1	73.4	204	229.7	104.7
25	28.1	13.5	85	95.7	43.4	145	163.2	73.9	205	230.8	105.3
26	29.3	14.0	86	96.8	43.9	146	164.4	74.4	206	231.9	105.8
27	30.4	14.5	87	97.9	44.4	147	165.5	74.9	207	233.0	106.3
28	31.5	15.0	88	99.1	44.9	148	166.6	75.5	208	234.2	106.8
29	32.7	15.5	89	100.2	45.4	149	167.7	76.0	209	235.3	107.4
30	33.8	16.0	90	101.3	45.9	150	168.9	76.5	210	236.4	107.9
31	34.9	16.5	91	102.4	46.4	151	170.0	77.0	211	237.6	108.4
32	36.0	17.0	92	103.6	46.9	152	171.1	77.5	212	238.7	109.0
33	37.2	17.5	93	104.7	47.4	153	172.3	78.1	213	239.8	109.5
34	38.3	18.0	94	105.8	47.9	154	173.4	78.6	214	240.9	110.0
35	39.4	18.5	95	107.0	48.4	155	174.5	79.1	215	242.1	110.6
36	40.5	18.9	96	108.1	48.9	156	175.6	79.6	216	243.2	111.1
37	41.7	19.4	97	109.2	49.4	157	176.8	80.1	217	244.3	111.6
38	42.8	19.9	98	110.3	49.9	158	177.9	80.7	218	245.4	112.1
39	43.9	20.4	99	111.5	50.4	159	179.0	81.2	219	246.6	112.7
40	45.0	20.9	100	112.6	50.9	160	180.1	81.7	220	247.7	113.2
41	46.2	21.4	101	113.7	51.4	161	181.3	82.2	221	248.7	113.7
42	47.3	21.9	102	114.8	51.9	162	182.4	82.7	222	249.9	114.3
43	48.4	22.4	103	116.0	52.4	163	183.5	83.3	223	251.0	114.8
44	49.5	22.9	104	117.1	52.9	164	184.6	83.8	224	252.4	115.3
45	50.7	23.4	105	118.2	53.5	165	185.8	84.3	225	253.3	115.9
46	51.8	23.9	106	119.3	54.0	166	186.9	84.8	226	254.4	116.4
47	52.9	24.4	107	120.5	54.5	167	188.0	85.3	227	255.6	116.9
48	54.0	24.9	108	121.6	55.0	168	189.1	85.9	228	256.7	117.4
49	55.2	25.4	109	122.7	55.5	169	190.3	86.4	229	257.8	118.0
50	56.3	25.9	110	123.8	56.0	170	191.4	86.9	230	258.9	118.5
51	57.4	26.4	111	125.0	56.5	171	192.5	87.4	231	260.1	119.0
52	58.5	26.9	112	126.1	57.0	172	193.6	87.9	232	261.2	119.6
53	59.7	27.4	113	127.2	57.5	173	194.8	88.5	233	262.3	120.1
54	60.8	27.9	114	128.3	58.0	174	195.9	89.0	234	263.4	120.7
55	61.9	28.4	115	129.6	58.6	175	197.0	89.5	235	264.6	121.2
56	63.0	28.8	116	130.6	59.1	176	198.1	90.0	236	265.7	121.7
57	64.2	29.3	117	131.7	59.6	177	199.3	90.5	237	266.8	122.3
58	65.3	29.8	118	132.8	60.1	178	200.4	91.1	238	268.0	122.8
59	66.4	30.3	119	134.0	60.6	179	201.5	91.6	239	269.1	123.4
60	67.6	30.8	120	135.1	61.1	180	202.6	92.1	240	270.2	123.9
61	68.7	31.3	121	136.2	61.6	181	203.8	92.6	241	271.3	124.4
62	69.8	31.8	122	137.4	62.1	182	204.9	93.1	242	272.5	125.0
63	70.9	32.3	123	138.5	62.6	183	206.0	93.7	243	273.6	125.5
64	72.1	32.8	124	139.6	63.1	184	207.1	94.2	244	274.7	126.0
65	73.2	33.3	125	140.7	63.7	185	208.3	94.7	245	275.8	126.6
66	74.3	33.8	126	141.9	64.2	186	209.4	95.2	246	277.0	127.1
67	75.4	34.3	127	143.0	64.7	187	210.5	95.7	247	278.1	127.6
68	76.6	34.8	128	144.1	65.2	188	211.7	96.3	248	279.2	128.1
69	77.7	35.3	129	145.2	65.7	189	212.8	96.8	249	280.3	128.7
70	78.8	35.8	130	146.4	66.2	190	213.9	97.3	250	281.5	129.2

*Allihn's table for the determination of dextrose—Continued.*

Milli-grams of copper.	Milli-grams of cuprous oxid.	Milli-grams of dextrose.	Milli-grams of copper.	Milli-grams of cuprous oxid.	Milli-grams of dextrose.	Milli-grams of copper.	Milli-grams of cuprous oxid.	Milli-grams of dextrose.	Milli-grams of copper.	Milli-grams of cuprous oxid.	Milli-grams of dextrose.
251	282.6	129.7	305	343.4	159.3	359	404.2	189.4	413	465.0	220.4
252	283.7	130.3	306	344.5	159.8	360	405.3	190.0	414	466.1	221.0
253	284.8	130.8	307	345.6	160.4	361	406.4	190.6	415	467.2	221.6
254	286.0	131.4	308	346.8	160.9	362	407.6	191.1	416	468.4	222.2
255	287.1	131.9	309	347.9	161.5	363	408.7	191.7	417	469.5	222.8
256	288.2	132.4	310	349.0	162.0	364	409.8	192.3	418	470.6	223.3
257	289.3	133.0	311	350.1	162.6	365	410.9	192.9	419	471.8	223.9
258	290.5	133.5	312	351.3	163.1	366	412.1	193.4	420	472.9	224.5
259	291.6	134.1	313	352.4	163.7	367	413.2	194.0	421	474.0	225.1
260	292.7	134.6	314	353.5	164.2	368	414.3	194.6	422	475.6	225.7
261	293.8	135.1	315	354.6	164.8	369	415.4	195.1	423	476.2	226.3
262	295.0	135.7	316	355.8	165.3	370	416.6	195.7	424	477.4	226.9
263	296.1	136.2	317	356.9	165.9	371	417.7	196.3	425	478.5	227.5
264	297.2	136.8	318	358.0	166.4	372	418.8	196.8	426	479.6	228.0
265	298.3	137.3	319	359.1	167.0	373	420.0	197.4	427	480.7	228.6
266	299.5	137.8	320	360.3	167.5	374	421.1	198.0	428	481.9	229.2
267	300.6	138.4	321	361.4	168.1	375	422.2	198.6	429	483.0	229.8
268	301.7	138.9	322	362.5	168.6	376	423.3	199.1	430	484.1	230.4
269	302.8	139.5	323	363.7	169.2	377	424.5	199.7	431	485.3	231.0
270	304.0	140.0	324	364.8	169.7	378	425.6	200.3	432	486.4	231.6
271	305.1	140.6	325	365.9	170.3	379	426.7	200.8	433	487.5	232.2
272	306.2	141.1	326	367.0	170.9	380	427.8	201.4	434	488.6	232.8
273	307.3	141.7	327	368.2	171.4	381	429.0	202.0	435	489.7	233.4
274	308.5	142.2	328	369.3	172.0	382	430.1	202.5	436	490.9	233.9
275	309.6	142.8	329	370.4	172.5	383	431.2	203.1	437	492.0	234.5
276	310.7	143.3	330	371.5	173.1	384	432.3	203.7	438	493.1	235.1
277	311.9	143.9	331	372.7	173.7	385	433.5	204.3	439	494.3	235.7
278	313.0	144.4	332	373.8	174.2	386	434.6	204.8	440	495.4	236.3
279	314.1	145.0	333	374.9	174.8	387	435.7	205.4	441	496.5	236.9
280	315.2	145.5	334	376.0	175.3	388	436.8	206.0	442	497.6	237.5
281	316.4	146.1	335	377.2	175.9	389	438.0	206.5	443	498.8	238.1
282	317.5	146.6	336	378.3	176.5	390	439.1	207.1	444	499.9	238.7
283	318.6	147.2	337	379.4	177.0	391	440.2	207.7	445	501.0	239.3
284	319.7	147.7	338	380.5	177.6	392	441.3	208.3	446	502.1	239.8
285	320.9	148.3	339	381.7	178.1	393	442.4	208.8	447	503.2	240.4
286	322.0	148.8	340	382.8	178.7	394	443.6	209.4	448	504.4	241.0
287	323.1	149.4	341	383.9	179.3	395	444.7	210.0	449	505.5	241.6
288	324.2	149.9	342	385.0	179.8	396	445.9	210.6	450	506.6	242.2
289	325.4	150.5	343	386.2	180.4	397	447.0	211.2	451	507.8	242.8
290	326.5	151.0	344	387.3	180.9	398	448.1	211.7	452	508.9	243.4
291	327.4	151.6	345	388.4	181.5	399	449.2	212.3	453	510.0	244.0
292	328.7	152.1	346	389.6	182.1	400	450.3	212.9	454	511.1	244.6
293	329.9	152.7	347	390.7	182.6	401	451.5	213.5	455	512.3	245.2
294	331.0	153.2	348	391.8	183.2	402	452.6	214.1	456	513.4	245.7
295	332.1	153.8	349	392.9	183.7	403	453.7	214.6	457	514.5	246.3
296	333.3	154.3	350	394.0	184.3	404	454.8	215.2	458	515.6	246.9
297	334.4	154.9	351	395.2	184.9	405	456.0	215.8	459	516.8	247.5
298	335.5	155.4	352	396.3	185.4	406	457.1	216.4	460	517.9	248.1
299	336.6	156.0	353	397.4	186.0	407	458.2	217.0	461	519.0	248.7
300	337.8	156.5	354	398.6	186.6	408	459.4	217.5	462	520.1	249.3
301	338.9	157.1	355	399.7	187.2	409	460.5	218.1	463	521.3	249.9
302	340.0	157.6	356	400.8	187.7	410	461.6	218.7			
303	341.1	158.2	357	401.9	188.3	411	462.7	219.3			
304	342.3	158.7	358	403.1	188.9	412	463.8	219.9			

## (c) METHODS FOR DETERMINATION OF COPPER CONTAINED IN THE PRECIPITATE OF CUPROUS OXID.—PROVISIONAL.

(For Low's volumetric method see Appendix, p. 241.)

## (1) REDUCTION IN HYDROGEN.

Filter the cuprous oxid immediately, through a weighed filtering tube made of hard glass, using suction. Support the asbestos film in the filtering tube with a perforated disk or cone of platinum, and wash free from loose fibers

before weighing; moisten previous to the filtration. Provide the tube with a detachable funnel during the filtration, so that none of the precipitate accumulates near the top, where it could be removed by the cork used during the reduction of the cuprous oxid. Transfer all the precipitate to the filter and thoroughly wash with hot water, following the water by alcohol and ether successively. After being dried, connect the tube with an apparatus for supplying a continuous current of dry hydrogen, gently heat until the cuprous oxid is completely reduced to the metallic state, cool in the current of hydrogen, and weigh. If preferred, a gooch crucible may be used for the filtration.

#### (2) ELECTROLYTIC DEPOSITION FROM SULPHURIC ACID SOLUTION.

Filter the cuprous oxid in a gooch, wash the beaker and precipitate thoroughly with hot water without any effort to transfer the precipitate to the filter. Wash the asbestos film and the adhering cuprous oxid into the beaker by means of hot dilute nitric acid. After the copper is all in solution, refilter through a gooch with a thin film of asbestos and wash thoroughly with hot water. Add 10 cc of dilute sulphuric acid, containing 200 cc of sulphuric acid (specific gravity 1.84) per liter, and evaporate the filtrate on the steam bath until the copper salt has largely crystallized. Heat carefully on a hot plate or over a piece of asbestos board until the evolution of white fumes shows that the excess of nitric acid is removed. Add from 8 to 10 drops of nitric acid (specific gravity, 1.42) and rinse into a platinum dish of from 100 to 125 cc capacity. Precipitate the copper by electrolysis. Wash thoroughly with water before breaking the current, remove the dish from the circuit, wash with alcohol and ether successively, dry at about 50° C., and weigh. If preferred, the electrolysis can be conducted in a beaker, the copper being deposited upon a weighed platinum cylinder.

#### (3) ELECTROLYTIC DEPOSITION FROM SULPHURIC AND NITRIC ACID SOLUTION.

Filter and wash as under (2). Transfer the asbestos film from the crucible to the beaker by means of a glass rod and rinse the crucible with about 30 cc of a boiling mixture of dilute sulphuric and nitric acids, containing 65 cc of sulphuric acid (specific gravity, 1.84) and 50 cc of nitric acid (specific gravity, 1.42) per liter. Heat and agitate until solution is complete; filter and electrolyze as under (2).

#### (4) ELECTROLYTIC DEPOSITION FROM NITRIC ACID SOLUTION.

Filter and wash as under (2). Transfer the asbestos film and adhering oxid to the beaker. Dissolve the oxid still remaining in the crucible by means of 2 cc of nitric acid (specific gravity, 1.42), adding it with a pipette and receiving the solution in the beaker containing the asbestos film. Rinse the crucible with a jet of water, allow the rinsings to flow into the beaker. Heat the contents of the beaker until the copper is all in solution, filter, dilute the filtrate to a volume of 100 cc or more, and electrolyze. When a nitrate solution is electrolyzed, the first washing of the deposit should be made with water acidulated with sulphuric acid, in order that the nitric acid may be all removed before the current is interrupted.

#### (5) VOLUMETRIC PERMANGANATE METHOD.

Filter and wash the cuprous oxid as described for method (2). Transfer the asbestos film to the beaker, add about 30 cc of hot water, and heat the precipitate and asbestos thoroughly. Rinse the crucible with 50 cc of a hot saturated

solution of ferric sulphate in 20 per cent sulphuric acid, receiving the rinsings in the beaker containing the precipitate. After the cuprous oxid is dissolved, wash the solution into a large Erlenmeyer flask and immediately titrate with a standard solution of potassium permanganate. One cc of the permanganate solution should equal 0.010 gram of copper. In order to determine the strength of this solution, make six or more determinations with the same sugar solution, titrating one-half of the precipitate obtained, and determining the copper in the others by electrolysis. The average weight of copper obtained by electrolysis, divided by the average number of cubic centimeters of permanganate solution required for the titration, is equal to the weight of copper equivalent to 1 cc of the standard permanganate solution. A solution standardized with iron or oxalic acid will give too low results.

#### (6) DIRECT WEIGHING OF CUPROUS OXID.

Prepare a gooch with an asbestos felt one-fourth of an inch thick. First thoroughly wash the asbestos with water to remove small particles, then follow successively with 10 cc of alcohol and 10 cc of ether, and dry the crucible and contents thirty minutes in a water oven at the temperature of boiling water.

Collect the precipitated cuprous oxid on the felt as usual, thoroughly wash with hot water, then with 10 cc of alcohol, and finally with 10 cc of ether. Dry the precipitate thirty minutes in a water oven at the temperature of boiling water; cool and weigh. The weight of cuprous oxid multiplied by 0.8883 gives the weight of metallic copper.

### 8. Starch.

#### (a) DIRECT ACID HYDROLYSIS (MODIFIED SACHSSE METHOD <sup>a</sup>).—OFFICIAL.

Stir a convenient quantity of the sample (representing from 2.5 to 3 grams of the dry material) in a beaker with 50 cc of water for an hour. Transfer to a filter and wash with 250 cc of water. Heat the insoluble residue for two and a half hours with 200 cc of water and 20 cc of hydrochloric acid (specific gravity 1.125) in a flask provided with a reflux condenser. Cool, and nearly neutralize with sodium hydroxid. Complete the volume to 250 cc, filter, and determine the dextrose in an aliquot of the filtrate as directed in (b) (2), page 49. The weight of the dextrose obtained multiplied by 0.90 gives the weight of starch.

#### (b) DIASTASE METHOD WITH SUBSEQUENT ACID HYDROLYSIS.—PROVISIONAL.

##### (1) PREPARATION OF MALT EXTRACT.

Digest 10 grams of fresh, finely ground malt two or three hours at ordinary temperature with 200 cc of water and filter. Determine the amount of dextrose in a given quantity of the filtrate after boiling with acid, etc., as in the starch determination, and make the proper correction in the subsequent determination.

##### (2) DETERMINATION.

Extract a convenient quantity of the substance (ground to an impalpable powder and representing from 4 to 5 grams of the dry material) on a hardened filter with five successive portions of 10 cc of ether; wash with 150 cc of 10

<sup>a</sup> In this method there will be included as starch the pentosans and other carbohydrate bodies present which suffer hydrolysis and conversion into reducing sugars on boiling with hydrochloric acid.

per cent alcohol and then with a little strong alcohol. Place the residue in a beaker with 50 cc of water, immerse the beaker in a boiling water bath, and stir constantly for fifteen minutes or until all the starch is gelatinized; cool to 55° C., add 20 cc of malt extract, and maintain at this temperature for an hour. Heat again to boiling for a few minutes, cool to 55° C., add 20 cc of malt extract, and maintain at this temperature for one hour or until a microscopic examination of the residue with iodine shows no starch. Cool and make up directly to 250 cc; filter. Place 200 cc of the filtrate in a flask with 20 cc of hydrochloric acid (sp. gr., 1.125); connect with a reflux condenser and heat in a boiling water bath for two and one-half hours. Cool, nearly neutralize with sodium hydroxid, and make up to 500 cc. Mix the solution well, pour through a dry filter, and determine the dextrose in an aliquot as directed on page 49. The weight of the dextrose obtained multiplied by 0.90 gives the weight of starch.

### 9. Pentosans.—Provisional.

#### (a) PREPARATION OF REAGENTS.

##### (1) QUALITATIVE TEST OF THE PURITY OF THE PHLOROGLUCOL.

Dissolve a small quantity of the phloroglucol in a few drops of acetic anhydride, heat almost to boiling, and add a few drops of concentrated sulphuric acid. A violet color indicates the presence of diresorcol. A phloroglucol which gives more than a faint coloration may be purified by the following method:

##### (2) PURIFICATION OF PHLOROGLUCOL.

Heat in a beaker about 300 cc of hydrochloric acid (specific gravity, 1.06) and 11 grams of commercial phloroglucol, added in small quantities at a time, stirring constantly until it has almost entirely dissolved. Some impurities may resist solution, but it is unnecessary to dissolve them. Pour the hot solution into a sufficient quantity of the same hydrochloric acid (cold) to make the volume 1,500 cc. Allow it to stand at least overnight—better several days—to allow the diresorcol to crystallize out, and filter immediately before using. The solution may turn yellow, but this does not interfere with its usefulness. In using it, add the volume containing the required amount to the distillate.

#### (b) DETERMINATION.

Place a quantity of the material, chosen so that the weight of phloroglucol obtained shall not exceed 0.300 gram, in a flask, together with 100 cc of 12 per cent hydrochloric acid (specific gravity, 1.06), and several pieces of recently heated pumice stone. Place the flask on a wire gauze, connect with a condenser, and heat, rather gently at first, and so regulate as to distil over 30 cc in about ten minutes, the distillate passing through a small filter paper. Replace the 30 cc driven over by a like quantity of the dilute acid added by means of a separatory funnel in such a manner as to wash down the particles adhering to the sides of the flask, and continue the process until the distillate amounts to 360 cc. To the completed distillate gradually add a quantity of phloroglucol (purified if necessary) dissolved in 12 per cent hydrochloric acid and thoroughly stir the resulting mixture. The amount of phloroglucol used should be about double that of the furfural expected. The solution first turns yellow, then green, and very soon an amorphous greenish precipitate appears, which grows rapidly darker, till it finally becomes almost black. Make the solution up to 400 cc with 12 per cent hydrochloric acid, and allow to stand over night.

Filter the amorphous black precipitate into a tared gooch crucible through an asbestos felt, wash carefully with 150 cc of water in such a way that the water is not entirely removed from the crucible until the very last, then dry for four hours at the temperature of boiling water, cool and weigh, in a weighing bottle, the increase in weight being reckoned as phloroglucid. To calculate the furfural, pentose, or pentosan from the phloroglucid, use the following formulas given by Kröber:

(a) For weight of phloroglucid "a" under 0.03 grams.

$$\text{Furfural} = (a + 0.0052) \times 0.5170.$$

$$\text{Pentoses} = (a + 0.0052) \times 1.0170.$$

$$\text{Pentosans} = (a + 0.0052) \times 0.8949.$$

(b) For weight of phloroglucid "a" over 0.300 gram.

$$\text{Furfural} = (a + 0.0052) \times 0.5180.$$

$$\text{Pentoses} = (a + 0.0052) \times 1.0026.$$

$$\text{Pentosans} = (a + 0.0052) \times 0.8824.$$

For weight of phloroglucid "a" from 0.03 to 0.300 grams use Kröber's table<sup>a</sup> or the following formulas:<sup>b</sup>

$$\text{Furfural} = (a + 0.0052) \times 0.5185.$$

$$\text{Pentoses} = (a + 0.0052) \times 1.0075.$$

$$\text{Pentosans} = (a + 0.0052) \times 0.8866.$$

#### 10. Galactan.—Provisional.

Extract a convenient quantity of the substance, representing from 2.5 to 3 grams of the dry material, on a hardened filter with five successive portions of 10 cc of ether, place the extracted residue in a beaker about 5.5 cm in diameter and 7 cm deep, together with 60 cc of nitric acid of 1.15 specific gravity, and evaporate the solution to exactly one-third its volume in a water bath at a temperature of 94° to 96° C. After standing twenty-four hours, add 10 cc of water to the precipitate, and allow it to stand another twenty-four hours. The mucic acid has in the meantime crystallized, but it is mixed with considerable material only partially oxidized by the nitric acid. Filter the solution therefore through filter paper, wash with 30 cc of water to remove as much of the nitric acid as possible, and replace the filter and contents in the beaker. Add 30 cc of ammonium carbonate solution, consisting of 1 part ammonium carbonate, 19 parts water, and 1 part strong ammonium hydroxid, and heat the mixture on a water bath, at 80° C., for fifteen minutes, with constant stirring. The ammonium carbonate takes up the mucic acid, forming the soluble mucate of ammonia. Then wash the filter paper and contents several times with hot water by decantation, passing the washings through a filter paper, to which finally transfer the material and thoroughly wash. Evaporate the filtrate to dryness over a water bath, avoiding unnecessary heating which causes decomposition, add 5 cc of nitric acid of 1.15 specific gravity, thoroughly stir the mixture and allow to stand for thirty minutes. The nitric acid decomposes the ammonium mucate, precipitating the mucic acid; collect this on a tared filter or gooch, wash with from 10 to 15 cc of water, then with 60 cc of alcohol, and a number of times with ether, dry at the temperature of boiling water for

<sup>a</sup> J. Landw., 1900, 48: 379. See p. 226 of this bulletin.

<sup>b</sup> These factors were calculated from Kröber's tables by C. A. Browne.

three hours, and weigh. Multiply mucic acid by 1.33, which gives galactose, and multiply this product by 0.9 which gives galactan.

### 11. Crude Fiber.—Official.

#### (a) PREPARATION OF REAGENTS.

Prepare solutions of sulphuric acid and sodium hydroxid of exactly 1.25 per cent strength, determined by titration.

#### (b) DETERMINATION.

Extract a quantity of the substance representing about 2 grams of the dry material with ordinary ether, or use residue from the determination of the ether extract. To this residue in a 500 cc flask add 200 cc of boiling 1.25 per cent sulphuric acid; connect the flask with an inverted condenser, the tube of which passes only a short distance beyond the rubber stopper into the flask. Boil at once and continue the boiling for thirty minutes. A blast of air conducted into the flask may serve to reduce the frothing of the liquid. Filter through linen, asbestos, or glass wool, wash with boiling water until the washings are no longer acid; rinse the substance back into the flask with 200 cc of a boiling 1.25 cc per cent solution of sodium hydroxid free, or nearly so, of sodium carbonate; boil at once and continue the boiling for thirty minutes in the same manner as directed above for the treatment with acid. Filter at once rapidly, wash with boiling water until the washings are neutral, dry at 110° C until it ceases to lose weight; weigh, incinerate completely, and weigh again. The loss of weight is considered to be crude fiber.



## **VII. METHODS FOR THE ANALYSIS OF CATTLE FOODS.**

### **1. Preparation of Sample.—Official.**

Grind the sample so that it will pass through a sieve having circular holes 1 mm in diameter. In case the sample can not be ground reduce it to as fine a state as possible.

### **2. Moisture.—Official.**

Dry the substance according to the directions given under "VI. General Methods," page 38.

### **3. Ash.—Official.**

Determine as directed under "VI. General Methods," page 38.

### **4. Nitrogenous Bodies.**

#### **(a) CRUDE PROTEIN OR TOTAL NITROGENOUS BODIES.—OFFICIAL.**

Determine nitrogen by the Kjeldahl or Gunning method, as directed under "I. Fertilizers," page 5, and multiply the result by 6.25.

#### **(b) PURE PROTEIN OR ALBUMINOID BODIES.—OFFICIAL.**

Determine the albuminoid nitrogen according to the directions given under "VI. General Methods," page 38, and multiply the result by 6.25.

#### **(c) AMIDO NITROGEN.—OFFICIAL.**

Subtract the amount of albuminoid nitrogen from the amount of total nitrogen to obtain the amido nitrogen.

### **5. Crude Fat or Ether Extract.—Official.**

Determine by the direct method under "VI. General Methods," page 39.

### **6. Sugars.**

#### **(a) PREPARATION OF SOLUTION.—PROVISIONAL.**

Weigh into a flask or bottle, suitable for stirring or shaking, 10 to 20 grams of the material, depending upon the amount of soluble carbohydrates present. Add 250 cc of ice-cold water, less the volume of water present as moisture in the material, and stir or shake for four hours. If enzymatic action is feared, the extraction should be made at a low temperature, preferably by surrounding the extraction flask with broken ice; or extract at ordinary temperature with 40 to 50 per cent alcohol. If there is present in the material much soluble substance, correction should also be made for the increase in volume due to solution. If necessary for clear filtration, add from 5 to 10 cc of alumina cream, prepared as directed under "VI. General Methods," page 40, just before filter-

ing. The volume of alumina cream to be added must be taken into account in determining the amount of water used for the extraction. After the extraction filter immediately, pouring back upon the filter the first portions of cloudy filtrate until the filtrate is clear. To free from soluble impurities add sufficient normal lead acetate solution to 200 cc of the filtrate to precipitate all impurities, make up to 250 cc, and filter. Remove the excess of lead by means of anhydrous sodium carbonate or anhydrous sodium sulphate, followed in the latter case by a small amount of anhydrous sodium carbonate, care being taken not to use an excess. Filter again and use the clear filtrate for the following determinations:

(b) REDUCING SUGAR.

(Calculated as dextrose, or invert sugar.)

Determine dextrose in a 50 cc aliquot of the clear filtrate by Allihn's method ("VI. General Methods," p. 49). Multiply the amount of dextrose by the factor 1.044 to obtain the equivalent in invert sugar.

(c) SUCROSE.

Invert a 50 cc aliquot of the clear filtrate according to one of the methods for the inversion of sucrose given under optical methods for determining sucrose ("VI. General Methods," p. 40). Determine the total reducing sugar and calculate as invert sugar according to (b). The total invert sugar less the invert sugar determined under (b) gives the true invert sugar from sucrose, and this multiplied by 0.95 gives sucrose.

7. Starch.

Determine according to the directions given under "VI. General Methods," page 53, section 8.

8. Pentosans.

Determine according to the directions given under "VI. General Methods," page 54, section 9.

9. Galactan.

Determine according to the directions given under "VI. General Methods," page 55, section 10.

10. Crude Fiber.

Determine in 2 grams of the air-dry material according to the directions given under "VI. General Methods," page 56, section 11.

## VIII. METHODS FOR THE ANALYSIS OF CEREAL FOODS.

[In preparation.]

## IX. METHODS FOR THE ANALYSIS OF CANNED VEGETABLES.— PROVISIONAL.

### 1. Physical Examination.

A careful examination of this character is often of material value in detecting inferior quality with certain classes of vegetables. Upon opening a can, carefully note the appearance of the contents as to quality, color, and size. Any undue corrosion or blackening of the walls of the can should also be observed. In the case of mushrooms and capers no further examination is necessary, as a rule, except the detection of sulphites in the former and copper in the latter.

The most common species of mushrooms found upon the market is *Agaricus campestris*, although different varieties of *Boletus* are occasionally found. The latter, particularly, are susceptible to attack by larvæ, and, except in a fresh state, are seldom free from them. These larvæ may readily be seen with the naked eye or by use of a small hand lens. Many of the mushrooms on the market are of inferior quality and consist largely of old and broken fragments of tops and stems; occasionally diseased fungi are to be found in the inferior grades. Owing to the nature of this vegetable, only the fresh, healthy specimens should be passed as edible.

Capers are but little liable to adulteration. Owing to their green color, it is always advisable to make a qualitative test for copper.

Olives are to be judged by general appearance and by taste. Gherkins and mixed pickles, while not strictly included in this class of foods, are considered here with olives for the sake of completeness; these also are to be judged largely macroscopically and by taste. Copper is sometimes used to produce a bright green color. When mustard is used with mixed pickles, turmeric is frequently added as a coloring agent. It is also advisable to test for anilin dyes if turmeric is not detected.

### 2. Preparation of the Sample.

Weigh the full can; open, pour off the liquid portion, and reweigh the can; then empty out the solid contents of the can and weigh again. From these weights estimate the percentage of liquid and solid contents. Then thoroughly grind the entire contents of the can either in a mortar or by means of a food chopper; mix thoroughly and preserve in a glass-stoppered bottle for analysis. Unless the analysis is to be completed within a reasonably short time, it is best to dry the entire sample after the determination of moisture is made.

After thorough drying, expose the material to the air for several hours, or until it becomes air dry. A second moisture determination is necessary with this procedure.

### 3. Total and Volatile Acids.

It is occasionally desirable to determine total acids in tomatoes and catsups, and volatile acids in the latter. For this purpose use the methods described

under "XIII. Wine," paragraphs 12 and 13, page 85. Express fixed acids as citric (1 cc of tenth-normal alkali equals 0.0070 gram of citric acid). Express volatile acids as acetic (1 cc of tenth-normal alkali equals 0.0060 gram of acetic acid).

#### 4. Detection of Saccharin.

Proceed as directed under "XXVII. Food Preservatives," page 182.

#### 5. Detection of Preservatives.

Proceed as directed under "XXVII. Food Preservatives," page 179.

#### 6. Detection of Coloring Matters.

##### (a) IN TOMATOES AND CATSUPS.

Extract the color from the dried pulp with alcohol, after acidifying with hydrochloric acid, and filter. Eosin gives a characteristic fluorescent filtrate. Dilute the filtrate with water, extract with amyl alcohol, and make dyeing tests. Cochineal, if present, is in the form of a lake and will require strong hydrochloric acid to decompose it. After extraction with amyl alcohol it may be tested with uranium acetate. (See also p. 190 under "XXVIII. Coloring Matter.")

##### (b) IN PEAS, BEANS, GHERKINS, ETC.

Copper salts are most commonly employed in this class of goods, although it is said that zinc is occasionally used. For the qualitative detection, ash from 15 to 20 grams of the sample, either with or without previous treatment with concentrated sulphuric acid (see "7. Heavy Metals," following), transfer the ash to a beaker, and treat with nitric acid; filter, make the filtrate alkaline with ammonium hydroxid, and, if a precipitate forms, filter again. Copper will be indicated by the blue color of the filtrate. If further test is desired, acidify with acetic acid and add potassium ferrocyanid.

##### (c) IN MIXED PICKLES, ETC.

Turmeric is frequently used and may be identified by the method given under "XXVIII. Coloring Matter" (p. 199).

#### 7. Heavy Metals.

##### (a) ALLEN'S METHOD MODIFIED.<sup>a</sup>

Treat 100 grams of the moist material, or 25 grams of the dried material, with about 5 cc of concentrated sulphuric acid and 2 cc of nitric acid. After foaming has ceased, add 3 grams of magnesium oxid and mix thoroughly. Ignite over a Bunsen burner or, preferably, in a muffle, until thoroughly charred. Grind in a mortar and again ignite to complete combustion. The addition of a few drops of nitric acid may be necessary toward the end to complete the operation. Add about 50 cc of hydrochloric acid (1:3) and heat to boiling or upon a steam bath for a half hour. Nearly neutralize the acid with sodium hydroxid, dilute to 150 cc with water, precipitate with hydrogen sulphid, and filter, after heating for a few moments upon a steam bath to facili-

<sup>a</sup> Bigelow and Munson, J. Amer. Chem. Soc. Proc., 1900, 22: 32.

tate the separation of the precipitated sulphids. Dry the precipitate and insoluble ash residue, and then fuse in a porcelain crucible with a mixture consisting of 1 gram each of sodium carbonate, potassium carbonate, and sulphur. Dissolve the fused mass with hot water and filter. Sulphids of lead and copper remain upon the filter. Acidify the filtrate with acetic acid to precipitate the tin sulphid. Collect the tin sulphid upon a filter. Wash thoroughly and then dissolve by the aid of heat in a concentrated solution of ferric chlorid. The reduced iron salt is then titrated with potassium dichromate. One cubic centimeter of tenth-normal potassium dichromate equals 0.00295 gram of tin. The determination of the tin by igniting and weighing as stannic oxid is unreliable, owing to the precipitation of appreciable amounts of silica dissolved by the mixed carbonates from the porcelain crucible. Determine the copper and lead, which remain as insoluble sulphids after the fusion, and the zinc, which remains in the original filtrate, according to the following method:

(b) MUNSON'S METHOD.

Treat 100 grams of the moist sample after evaporating to dryness, or 25 grams of the dry sample, in a 4-inch porcelain evaporating dish with sufficient concentrated sulphuric acid to thoroughly carbonize the mass. Gently heat over a Bunsen burner until all danger of foaming is past, which will require not more than three minutes; then transfer the dish to a muffle and keep it at a low red heat until all organic matter is destroyed. It is occasionally found necessary to add a few drops of nitric acid to completely destroy organic matter. When the material is completely ashed, allow the dish to cool; add 25 cc of hydrochloric acid (1:8) and evaporate on a water bath to dryness; take up with water and acidify with two or three drops of hydrochloric acid. Transfer to a beaker without filtering and treat with hydrogen sulphid. After heating upon a water bath for a few minutes the precipitate and the insoluble residue are collected upon a filter. The precipitate and residue may contain sulphids of tin, lead, and copper, and oxid of tin; the filtrate will contain any zinc that is present.

Fuse the sulphid precipitate and insoluble ash residue with about 3 grams of caustic soda in a silver crucible for a half hour to render soluble any insoluble tin compounds. Dissolve the mass with hot water and slightly acidify with hydrochloric acid. Again treat with hydrogen sulphid without filtering. By this treatment all the tin is thrown down as sulphid with the sulphids of copper and lead. Collect the precipitate upon a filter and wash thoroughly with hot water. The filtrate may be rejected. To separate the tin sulphid from those of copper and lead, wash several times upon the filter with separate portions of 10 cc of strong boiling ammonium sulphid. Usually 50 cc of ammonium sulphid will be found sufficient to completely dissolve all tin sulphid, but portions of the filtrate should be tested to prove this point. Acidify with hydrochloric acid to precipitate the tin sulphid, which, after standing for a few moments, is collected upon an ashless filter, ignited, and weighed as stannic oxid.

Treat the insoluble residue remaining from the ammonium sulphid washing with nitric acid, filter, wash, nearly neutralize the excess of mineral acid with ammonium hydroxid, and add ammonium acetate, as there is usually a small amount of iron present. If an iron salt is precipitated, filter, wash, and divide the filtrate for the determinations of copper and lead. In the absence of lead, copper may be determined electrolytically, or it may be titrated with potassium cyanid. Unless added as a coloring agent, copper will seldom be present in sufficient quantity to warrant its determination.

Precipitate lead with potassium chromate in an acetic acid solution; and weigh upon a tared filter as lead chromate.

Evaporate the filtrate from the hydrogen sulphid precipitate to about 60 cc; add bromin water to oxidize the iron salts and any remaining hydrogen sulphid. Boil off the excess of bromin and, unless the solution is distinctly yellow, add a few drops of concentrated solution of ferric chlorid to make it so. Nearly neutralize the mineral acid with ammonium hydroxid, and add ammonium acetate to precipitate iron phosphate and excess of iron. Filter and thoroughly wash the precipitate. To the filtrate, made distinctly acid with acetic acid and boiled, add hydrogen sulphid to precipitate zinc. Unless the zinc sulphid comes down white, it should be dissolved, again treated with ammonium acetate to remove traces of iron, and reprecipitated as sulphid. Finally collect the zinc sulphid upon an ashless filter, ignite, and weigh as zinc oxid.

### 8. Soaked Vegetables.

A class of canned vegetables commercially known as "soaked" goods is frequently found upon the market and constitutes the cheapest grade of vegetables sold. Peas, beans, and corn, or combinations of these three, are most often found in this class. The materials used for "soaked" products are the ordinary matured peas and beans, such as are used for seed or are sold dried upon the market, and corn that has passed the stage at which it can be supplied for the green market.

The composition of soaked vegetables probably varies but little from that of the younger products, yet they do not possess an equal value as a relish. In the mature vegetables the percentage of total solids is much higher than in the young and more succulent vegetables, and this condition holds true in the canned goods if only the solid contents of the can are considered.

Soaked peas and beans have the general appearance of the well-matured product. The cotyledons are well formed, firm, and mealy. With the pea the caulicle is particularly prominent, the process of soaking having started its development. In the case of corn, the kernel is plump and hard and less milky than the younger kernels. The succulence so characteristic of the green pea, bean, and corn is entirely lacking. From their nature it is difficult to apply specific tests to this class of goods, but a little practice will enable the analyst to detect them with reasonable certainty by inspection of their appearance and by the sense of taste.

## X. METHODS FOR THE ANALYSIS OF SACCHARINE PRODUCTS.

### 1. Preparation of Sample.—Provisional.

#### (a) MOLASSES, SIRUPS, HONEY, ETC.

Materials of this class must be thoroughly mixed, and in case any sugars have crystallized out these must be dissolved by gently heating before analysis. In the case of comb honey, cut across the top of the comb, if sealed, and separate completely from the comb by straining through a 40-mesh sieve.

#### (b) SEMIPLASTIC, SIRUPY, OR PASTY PRODUCTS.

Weigh 50 grams of the sample into a 250 graduated flask, mix thoroughly or dissolve, if soluble, in water and fill to the mark. Be sure that the solution is uniform, or, if insoluble material is present, that it is evenly mixed by shaking before taking aliquots for the various determinations.

#### (c) SUGAR AND CONFECTIONERY.

Materials of this class must be ground and thoroughly mixed to secure uniformity of sample.

### 2. Moisture.

#### (a) BY DRYING.

##### (1) IN SUGARS.—OFFICIAL.

Dry from 2 to 5 grams in a flat dish (nickel, platinum, or aluminum) at the temperature of boiling water for ten hours; cool in a desiccator and weigh; return to the oven and dry for an hour or until there is only a slight change in weight.

##### (2) IN MASSECUTES, MOLASSES, HONEYS, AND OTHER LIQUID AND SEMILIQUID PRODUCTS.—PROVISIONAL.

Prepare pumice stone in two grades of fineness. One of these should pass through a 1 mm sieve, while the other should be composed of particles too large for a millimeter sieve, but sufficiently small to pass through a sieve having meshes 6 mm in diameter. Make the determination in flat metallic dishes or in shallow, flat-bottom, weighing bottles. Place a layer of the fine pumice stone 3 mm in thickness over the bottom of the dish, and upon this place a layer of the coarse pumice stone from 6 to 10 mm in thickness. Dry the dish thus prepared and weigh. Dilute the sample with a weighed portion of water in such a manner that the diluted material shall contain from 20 to 30 per cent of dry matter. Weigh into the dish, prepared as described above, such a quantity of the diluted sample as will yield, approximately, 1 gram of dry



matter. Use a weighing bottle provided with a cork through which a pipette passes if this weighing can not be made with extreme rapidity. Place the dish in a water oven and dry to constant weight at the temperature of boiling water, making trial weighings at intervals of two hours. In case of materials containing much levulose or other readily decomposable substances, conduct the drying in vacuo at about 70° C.

(3) FOR DRYING MOLASSES WITH QUARTZ SAND.—PROVISIONAL.

In a flat-bottom dish place 6 or 7 grams of pure quartz sand and a short stirring rod. Dry thoroughly, cool in a desiccator, and weigh. Then add 3 or 4 grams of the molasses, mix with the sand, and dry at the temperature of boiling water for from eight to ten hours. Stir at intervals of an hour, then cool in a desiccator, and weigh. Stir, heat again in the water oven for an hour, cool, and weigh. Repeat heating and weighing until loss of water in one hour is not greater than 3 mg.

Before using, digest the pure quartz sand with strong hydrochloric acid, wash, dry, ignite, and keep in a stoppered bottle.

(b) AREOMETRIC METHODS.<sup>a</sup>—OFFICIAL.

(1) SPECIFIC GRAVITY, WATER, AND TOTAL SOLIDS BY MEANS OF A SPINDLE.

The density of juices, sirups, etc., is most conveniently determined by means of the Baumé or Brix hydrometer, preferably the latter, as the graduation of the scale gives close approximations to the percentages of total solids. The Brix spindle should be graduated to tenths. The range of degrees recorded by each individual spindle should be as limited as possible. The solution should be as nearly as practicable of the same temperature as the air at the time of reading, and if the variation from the temperature of the graduation of the spindle amounts to more than 1°, a compensation must be applied according to the table of corrections for temperature, page 67. Before taking the density of a juice it should be allowed to stand in the cylinder until all air bubbles have escaped.

In case the sample is too dense to determine the density directly, dilute a weighed portion with a weighed quantity of water, or dissolve a weighed portion and dilute to a known volume with water. In the first instance the per cent of total solids is calculated by the following formula:

$$\text{Per cent of solids in the undiluted material} = \frac{WS}{W}$$

S=per cent of solids in the diluted material.

W=weight of the diluted material.

w=weight of the sample taken for dilution.

When the dilution is made to a definite volume, the following formula is to be used:

$$\text{Per cent of solids in the undiluted material} = \frac{VDS}{W}$$

V=volume of the diluted solution.

D=specific gravity of the diluted solution.

S=per cent of solids in the diluted solution.

W=weight of the sample taken for dilution.

<sup>a</sup> This method does not apply to low-grade sugar products since materials high in salts give excessive percentages.

A table for the comparison of specific gravities  $\left(\frac{17.5^\circ}{17.5^\circ}\right)$ , degrees Brix (per cent by weight of sucrose) and degrees Baumé, is given below.

(2) SPECIFIC GRAVITY, WATER, AND TOTAL SOLIDS BY MEANS OF A PYCNOMETER.

When a more accurate determination of the per cent of solids, or of water, or of the specific gravity is desired, the determination should be made with a specific gravity bottle or pycnometer. When of too high density for a direct determination, the sample may be diluted, as described under (1).

*A table for the comparison of specific gravities, degrees Brix and degrees Baumé.*

Degree Brix or per cent by weight of sucrose.	Specific gravity.	Degree Baumé.	Degree Brix or per cent by weight of sucrose.	Specific gravity.	Degree Baumé.	Degree Brix or per cent by weight of sucrose.	Specific gravity.	Degree Baumé.
1.0	1.00388	0.6	33.0	1.14423	18.5	65.0	1.31989	35.6
2.0	1.00779	1.1	34.0	1.14915	19.05	66.0	1.32601	36.1
3.0	1.01173	1.7	35.0	1.15411	19.6	67.0	1.33217	36.6
4.0	1.01570	2.3	36.0	1.15911	20.1	68.0	1.33836	37.1
5.0	1.01970	2.8	37.0	1.16413	20.7	69.0	1.34460	37.6
6.0	1.02373	3.4	38.0	1.16920	21.2	70.0	1.35088	38.1
7.0	1.02779	4.0	39.0	1.17430	21.8	71.0	1.35720	38.6
8.0	1.03187	4.5	40.0	1.17943	22.3	72.0	1.36355	39.1
9.0	1.03599	5.1	41.0	1.18460	22.9	73.0	1.36995	39.6
10.0	1.04014	5.7	42.0	1.18981	23.4	74.0	1.37639	40.1
11.0	1.04431	6.2	43.0	1.19505	23.95	75.0	1.38287	40.6
12.0	1.04852	6.8	44.0	1.20033	24.5	76.0	1.38939	41.1
13.0	1.05276	7.4	45.0	1.20565	25.0	77.0	1.39595	41.6
14.0	1.05703	7.9	46.0	1.21100	25.6	78.0	1.40254	42.1
15.0	1.06133	8.5	47.0	1.21639	26.1	79.0	1.40918	42.6
16.0	1.06566	9.0	48.0	1.22182	26.6	80.0	1.41586	43.1
17.0	1.07002	9.6	49.0	1.22728	27.2	81.0	1.42258	43.6
18.0	1.07441	10.1	50.0	1.23278	27.7	82.0	1.42934	44.1
19.0	1.07884	10.7	51.0	1.23832	28.2	83.0	1.43614	44.6
20.0	1.08329	11.3	52.0	1.24390	28.8	84.0	1.44298	45.1
21.0	1.08778	11.8	53.0	1.24951	29.3	85.0	1.44986	45.5
22.0	1.09231	12.4	54.0	1.25517	29.8	86.0	1.45678	46.0
23.0	1.09686	13.0	55.0	1.26086	30.4	87.0	1.46374	46.5
24.0	1.10145	13.5	56.0	1.26658	30.9	88.0	1.47074	47.0
25.0	1.10607	14.1	57.0	1.27235	31.4	89.0	1.47778	47.5
26.0	1.11072	14.6	58.0	1.27816	31.9	90.0	1.48486	47.9
27.0	1.11541	15.2	59.0	1.28400	32.5	91.0	1.49199	48.5
28.0	1.12013	15.7	60.0	1.28989	33.0	92.0	1.49915	48.9
29.0	1.12488	16.3	61.0	1.29581	33.5	93.0	1.50635	49.4
30.0	1.12967	16.8	62.0	1.30177	34.0	94.0	1.51359	49.8
31.0	1.13449	17.4	63.0	1.30777	34.5	95.0	1.52087	50.3
32.0	1.13934	17.95	64.0	1.31381	35.1			

When the number expressing the specific gravity found by analysis falls between the numbers given in the above table, the exact equivalent in degrees Brix or Baumé is found by a simple calculation.

Example.—The pycnometer shows the specific gravity of a certain sirup to be 1.20909. The table shows that the corresponding degree Brix is between 45.0 and 46.0. Subtracting the specific gravity of a solution of 45° Brix from the corresponding figure for 46°, we have (expressing the specific gravities as whole numbers) 121,100—120,565=535, the difference in specific gravity for 1° Brix at this point in the table. Subtracting the specific gravity corresponding to 45° from the specific gravity found by analysis, we have 120,909—120,565=344;  $\frac{344}{535}=0.64$ , the fraction of 1° Brix more than 45°. The degree Brix, corresponding to a specific gravity of 1.20909, is therefore 45.64.

If the spindle reading or pycnometer determination be made at any other tem-

perature than 17.5° C., the result should be corrected by the use of the following table:

*Table for correction of the readings of the Brix spindle when the reading is made at other than the standard temperature, 17.5°.*

[For temperatures below 17.5° the correction is to be subtracted.]

Temperature.	Degree Brix of the solution.												
	0	5	10	15	20	25	30	35	40	50	60	70	75
°C.													
0	0.17	0.30	0.41	0.52	0.62	0.72	0.82	0.92	0.98	1.11	1.22	1.25	1.29
5	0.23	0.30	0.37	0.44	0.52	0.59	0.65	0.72	0.75	0.80	0.88	0.91	0.94
10	0.20	0.26	0.29	0.33	0.36	0.39	0.42	0.45	0.48	0.50	0.54	0.58	0.61
11	0.18	0.23	0.26	0.28	0.31	0.34	0.36	0.39	0.41	0.43	0.47	0.50	0.53
12	0.16	0.20	0.22	0.24	0.26	0.29	0.31	0.33	0.34	0.36	0.40	0.42	0.46
13	0.14	0.18	0.19	0.21	0.22	0.24	0.26	0.27	0.28	0.29	0.33	0.35	0.39
14	0.12	0.15	0.16	0.17	0.18	0.19	0.21	0.22	0.22	0.23	0.26	0.23	0.32
15	0.09	0.11	0.12	0.14	0.14	0.15	0.16	0.17	0.16	0.17	0.19	0.21	0.25
16	0.06	0.07	0.08	0.09	0.10	0.10	0.11	0.12	0.12	0.12	0.14	0.16	0.18
17	0.02	0.02	0.03	0.03	0.03	0.04	0.04	0.04	0.04	0.04	0.05	0.05	0.06
18	0.02	0.03	0.03	0.03	0.03	0.03	0.03	0.03	0.03	0.03	0.03	0.03	0.02
19	0.06	0.08	0.08	0.09	0.09	0.10	0.10	0.10	0.10	0.10	0.10	0.08	0.06
20	0.11	0.14	0.15	0.17	0.17	0.18	0.18	0.18	0.19	0.19	0.18	0.15	0.11
21	0.16	0.20	0.22	0.24	0.24	0.25	0.25	0.25	0.26	0.26	0.25	0.22	0.18
22	0.21	0.26	0.29	0.31	0.31	0.32	0.32	0.32	0.33	0.34	0.32	0.29	0.25
23	0.27	0.32	0.35	0.37	0.38	0.39	0.39	0.39	0.40	0.42	0.39	0.36	0.33
24	0.32	0.38	0.41	0.43	0.44	0.46	0.46	0.47	0.47	0.50	0.46	0.43	0.40
25	0.37	0.44	0.47	0.49	0.51	0.53	0.54	0.55	0.55	0.58	0.54	0.51	0.48
26	0.43	0.50	0.54	0.56	0.58	0.60	0.61	0.62	0.62	0.66	0.62	0.58	0.55
27	0.49	0.57	0.61	0.63	0.65	0.68	0.68	0.69	0.70	0.74	0.70	0.65	0.62
28	0.56	0.64	0.68	0.70	0.72	0.76	0.76	0.78	0.78	0.82	0.78	0.72	0.70
29	0.63	0.71	0.75	0.78	0.79	0.84	0.84	0.86	0.86	0.90	0.86	0.80	0.78
30	0.70	0.78	0.82	0.87	0.87	0.92	0.92	0.94	0.94	0.98	0.94	0.88	0.86
35	1.10	1.17	1.22	1.24	1.30	1.32	1.33	1.35	1.36	1.39	1.34	1.27	1.25
40	1.50	1.61	1.67	1.71	1.73	1.79	1.79	1.80	1.82	1.83	1.78	1.69	1.65
50	.....	2.65	2.71	2.74	2.78	2.80	2.80	2.80	2.80	2.79	3.70	2.56	2.51
60	.....	3.87	3.88	3.88	3.88	3.88	3.88	3.88	3.80	3.82	2.70	3.43	3.41
70	.....	5.17	5.18	5.20	5.14	5.13	5.10	5.08	5.06	4.90	4.72	4.47	4.35
80	.....	.....	6.62	6.59	6.54	6.46	6.38	6.30	6.26	6.06	5.82	5.50	5.33
90	.....	.....	8.26	8.16	8.06	7.97	7.83	7.71	7.58	7.30	6.96	6.58	6.37
100	.....	.....	10.01	9.87	9.72	9.56	9.39	9.21	9.03	8.64	8.22	7.76	7.42

Example.—A sugar solution shows a reading of 30.2° Brix at 30° C. To find the necessary correction for the conversion of this reading to the reading which would have been obtained if the observation had been made at 17.5° C., find the vertical column in the table headed 30° Brix, which is the nearest to the observed reading. Follow down this column until the number is reached which is opposite to the temperature of observation—in this case 30°. The number found, 0.92, is to be added to the observed reading.

### 3. Ash.

#### (a) DETERMINATION.—OFFICIAL.

##### (1) METHOD I.

Heat from 5 to 10 grams of sugar, molasses, maple products, or honey in a platinum dish<sup>a</sup> of from 50 to 100 cc capacity at 100° C. until the water is expelled, and then slowly over a flame until intumescence ceases. Then place the dish in a muffle and heat at low redness until a white ash is obtained.

For soluble ash digest the ash obtained as above with water, filter through a gooch, wash with hot water, and dry the residue at 100° C.

<sup>a</sup> If the substance contains tin or any other metal capable of uniting with platinum, a dish made of some other material must be used.

## (2) METHOD II. (Dropped by action in 1907.)

Use 50 mg of zinc oxid to 25 grams of molasses or 50 grams of sugar. Incorporate thoroughly by adding dilute alcohol and mixing; dry and ignite as above. Deduct the weight of zinc oxid used from the weight of the ash.

## (3) METHOD III.

Carbonize the mass at a low heat, dissolve the soluble salts with hot water, burn the residual mass as above, add the solution of soluble salts, and evaporate to dryness at 100° C., ignite gently, cool in a desiccator, and weigh.

## (4) METHOD IV.

Saturate the sample with sulphuric acid, dry, ignite gently, then burn in a muffle at low redness. Deduct one-tenth of the weight of the ash, then calculate the per cent.

## (5) METHOD V.

Thoroughly mix 5 grams of the material with a somewhat larger weight of pure quartz sand in a platinum dish; ignite in a muffle at a moderate red heat.

## (6) METHOD VI.

To avoid the correction of one-tenth, as proposed by Scheibler, and one-fifth, as proposed by Girard and Violette, when sugars are burned with sulphuric acid, Boyer suggests incineration with benzoic acid as giving the real quantity of mineral matter without correction.

Dissolve 25 grams of the benzoic acid in 100 cc of 90 per cent alcohol. Weigh 5 grams of the sugar in a capsule and moisten with 1 cc of water. Heat the capsule slowly to caramelize the sugar without carbonizing it; then add 2 cc of the benzoic acid solution and warm the capsule until all the alcohol is evaporated; raise the temperature until the sugar is converted into carbon. The decomposing benzoic acid produces abundant vapors which render the mass extremely porous, especially if a circular motion be imparted to the capsule. The slow heating is continued until all the benzoic acid is volatilized. The carbon obtained is voluminous and of a brilliant black color. The incineration is accomplished in a muffle at a low red heat. The capsule should be weighed quickly when taken from the desiccator, in order to avoid the absorption of water by the alkaline carbonates. Ammonium benzoate may be employed instead of benzoic acid, and the analyst should previously assure himself that neither the acid nor the ammonium salt leaves a residue on incineration. In addition to giving the mineral matter directly, this method permits the determination of its composition also.

## (b) QUANTITATIVE ANALYSIS OF THE ASH.—OFFICIAL.

Proceed as directed under "III. Inorganic Plant Constituents," page 21.

## (c) SOLUBLE AND INSOLUBLE ASH.—PROVISIONAL.

Ash the material according to Method I under the determination of ash, page 67, "3. (a)"; add water to the ash in the platinum dish, heat nearly to boiling, filter through ash-free filter paper, and wash with hot water until the filtrate and washings amount to about 60 cc. Return the filter paper and contents to the platinum dish, carefully ignite, and weigh. Compute percentages of water-insoluble ash and water-soluble ash.

## (d) ALKALINITY OF SOLUBLE ASH.—PROVISIONAL.

Allow the filtrate from the above determination to cool, then titrate with tenth-normal hydrochloric acid, using methyl orange as an indicator.

## (e) ALKALINITY OF INSOLUBLE ASH.—PROVISIONAL.

Add excess of tenth-normal hydrochloric acid (usually 10 to 15 cc) to the ignited insoluble ash in the platinum dish, heat to the point of boiling over an asbestos plate, allow to cool, and titrate excess of hydrochloric acid with tenth-normal sodium hydroxid, using methyl orange as an indicator.

Express the alkalinity in each case as the number of cubic centimeters of tenth-normal acid used on the ash of 1 gram of sample.

## (f) MINERAL ADULTERANTS IN ASH.

## (1) REDUCTION TO ASH.—PROVISIONAL.

Comparatively large quantities of saccharine products may be readily and quickly reduced to an ash for mineral examination without the troublesome frothing that ordinarily ensues in igniting at once with a free flame by proceeding as follows: <sup>a</sup>

Mix 100 grams of molasses, sirup, or honey, or of the confectionery solution (b) under "1. Preparation of Sample," p. 64, evaporated to a sirupy consistency, with about 35 grams of concentrated sulphuric acid in a large porcelain evaporating dish. Then pass an electric current through it while stirring by placing one platinum electrode in the bottom of the dish near one side and attaching the other to the lower end of the glass rod with which the contents are stirred. Begin with a current of about 1 ampère and gradually increase to 4.<sup>b</sup> In from ten to fifteen minutes the mass is reduced to a fine dry char, which may then be readily burnt to a white ash in the original dish over a free flame or in a muffle.

If an electric current is unavailable, treat in a large porcelain dish 100 grams of the saccharine solution to be ashed, which should be evaporated to a sirupy consistency if not already in such condition, with sufficient concentrated sulphuric acid to thoroughly carbonize the mass, after which ignite in the usual manner.

Among the suspected adulterants to be looked for in the ash are salts of tin, used in molasses to bleach or lighten the color; mineral pigments, such as chromate of lead in yellow confectionery and oxid of iron, the latter being sometimes used as an intensifier of or substitute for the natural color of chocolate.

(2) TIN IN MOLASSES <sup>c</sup> AND OTHER SACCHARINE PRODUCTS.<sup>d</sup>—PROVISIONAL.

Fuse the ash from a weighed portion of the sample with sodium hydroxid in a silver crucible, dissolve in water, and acidulate with hydrochloric acid; <sup>e</sup>

<sup>a</sup> Leach, Thirty-second Ann. Rept. Mass. State Board of Health, 1900, p. 653; reprint, p. 37. This method is preferred to the ordinary method of heating with sulphuric acid, especially in the case of molasses, because, if properly manipulated, it comes quietly into the form of a very finely divided char or powder, especially adapted for subsequent quick ignition.

<sup>b</sup> Modified from method of Budde and Schou for determining nitrogen electrolytically. Zts. anal. Chem., 1899, 38: 345.

<sup>c</sup> Leach, Thirty-second Ann. Rept. Mass. State Board of Health, 1899, p. 625; Hilger and Laband, Zts. Nahr. Genussm., 1899, 2: 795.

<sup>d</sup> This method is applicable also to condensed milk, canned goods, etc.

<sup>e</sup> See page 61, under "IX. Canned Vegetables," section 7.

filter and precipitate the tin from this solution with hydrogen sulphid; wash the precipitate on a filter and dissolve it in an excess of ammonium sulphid. Filter this solution into a tared platinum dish and deposit the tin directly in the dish by electrolysis, using a current of 0.05 ampère. This current may be readily reduced from an ordinary 110-volt street circuit by means of a series of lamps, or a rheostat may be improvised for this purpose, consisting of a long, vertical glass tube, sealed at the bottom, containing a column of dilute acid through which the current passes, the resistance being changed by varying the length of the acid column contained between two electrodes immersed therein, one of which is movable.<sup>a</sup>

#### 4. Nitrogen.—Provisional.

Determine in 5 grams of the sample by the Kjeldahl or Gunning methods, page 5, under "I. Fertilizers."

#### 5. Sucrose.—Provisional.

Calculate sucrose from the polarization<sup>b</sup> before and after inversion, as directed under "VI. General Methods," page 41. In case of confectionery containing insoluble matter employ the double-dilution method,<sup>c</sup> thus making due allowance for the volume of the precipitate, as follows: Use half the normal weight of the sample and make up the solution to 100 cc, employing the appropriate clarifier (subacetate of lead for dark-colored confectionery or molasses and alumina cream for light-colored confectionery and honey). Use the normal weight of the sample and make up a second solution with the clarifier to 100 cc. Filter and obtain direct polariscopic readings of both solutions. Invert each in the usual manner and obtain the invert readings of the two.

The true *direct* polarization of the sample is the product of the two direct readings divided by their difference.

The true *invert* polarization is the product of the two invert readings divided by their difference.

#### 6. Commercial Glucose in Molasses, Sirups, and Honey.<sup>d</sup> (Approximate Method).—Provisional.

It is manifestly impossible to determine with absolute accuracy the amount of commercial glucose present by reason of the varying amounts of dextrin, maltose, and dextrose present in the adulterant. It is possible, however, in sirups, in which the amount of invert sugar is so small as not to appreciably affect the result, to estimate approximately the amount of commercial glucose by the following formula:

$$G = \frac{(a-S) 100}{175}$$

<sup>a</sup> Wiley, Principles and Practice of Agricultural Analysis, 3: 152.

<sup>b</sup> All products, such as honeys, sirups, etc., which contain dextrose or other reducing sugars in the crystalline form or in supersaturated solution, exhibit the phenomenon of birotation. The constant rotation only should be employed in the Clerget formula, and to obtain this the solutions prepared for direct polarization should be allowed to stand over night before making the reading. In case it is desired to make the direct reading immediately the birotation may be destroyed by heating the neutral solution to boiling for a few minutes or by adding a few drops of strong ammonium hydroxid before completing the volume.

<sup>c</sup> Wiley and Ewell, Analyst, 1896, 21: 182.

<sup>d</sup> Leach, Thirty-second Ann. Rept. Mass. State Board of Health, 1900, p. 658; reprint, p. 42.

where G = per cent of commercial glucose, a = direct polarization, S = per cent of cane sugar. Express results in terms of commercial glucose polarizing  $+175^{\circ}$  V.

In substances which consist largely of invert sugar much more accurate results are attained by polarizing at  $87^{\circ}$  C. In a water-jacketed tube an inverted half-normal solution of the sample (13 grams) prepared as directed under "VI. General Methods," page 41, with the following exceptions: After inversion, cool, add a few drops of phenolphthalein and enough sodium hydroxid to neutralize; discharge the pink color with a few drops of dilute hydrochloric acid, add from 5 to 10 cc of alumina cream, and make up to the mark and filter. Multiply by 2 the reading at  $87^{\circ}$  C. in the 200 mm tube; multiply this result by 100 and divide by the factor 163 to express the glucose <sup>a</sup> in terms of glucose polarizing  $+175^{\circ}$  V.

#### 7. Reducing Sugars.—Provisional.

Determine either as dextrose or invert sugar as directed under "VI. General Methods," pages 42 and 49.

#### 8. Starch.—Provisional.

Measure gradually 25 cc of the 20 per cent solution or uniform mixture (1. Preparation of Sample, (b), p. 64) into a hardened filter or gooch crucible, or transfer by washing 5 grams of the finely powdered substance to the filter or gooch, and allow the residue on the filter to become air dried. Extract with 5 successive portions of 10 cc of ether, then wash with 150 cc of 10 per cent alcohol, and finally with 20 cc of strong alcohol. Transfer the residue to a large flask and determine starch as directed under "VI. General Methods," page 53.

#### 9. Ether Extract in Confectionery.—Provisional.

Pipette 25 cc of the 20 per cent mixture or solution (1. Preparation of Sample (b), p. 64) into a very thin, readily frangible, glass evaporating shell (*Hoffmeister's Schälchen*), containing 5 to 7 grams of freshly ignited asbestos fiber; or, if impossible to obtain thus a uniform sample, weigh out 5 grams of the mixed finely divided sample into a dish, and wash with water into the asbestos in the evaporating shell, using, if necessary, a small portion of the asbestos fiber on a stirring rod to transfer the last traces of the sample from dish to shell. Dry to constant weight at  $100^{\circ}$  C., after which cool, wrap loosely in smooth paper, and crush into rather small fragments between the fingers, carefully transferring the pieces with the aid of a camel's-hair brush to an extraction tube or a Schleicher and Schüll cartridge for fat extraction. A thin lead disk (bottle cap) may be substituted for the *Schälchen*. The disk may then be cut into small pieces and placed in the extraction tube. Extract with anhydrous ether or petroleum ether in a continuous-extraction apparatus for at least twenty-five hours. Transfer to a tared flask, evaporate the ether, dry in an oven at  $100^{\circ}$  C. to constant weight, and weigh.

If petroleum ether is employed, it should be purified by fractional distillation so that it boils between  $45^{\circ}$  and  $60^{\circ}$  C. and leaves absolutely no residue.

<sup>a</sup> Nearly all pure honeys are more or less dextro-rotatory at  $87^{\circ}$  C., according to the amount of natural dextrin present. The reading at  $87^{\circ}$  C., however, rarely exceeds  $20^{\circ}$  V. (26 grams per 100 cc), and then only in abnormal samples containing large quantities of honeydew or other exudations than the nectar of flowers. To distinguish between abnormal honeydew honeys and honeys adulterated with glucose, see König (*Zts. anal. Chem.*, 1895, 34: 1) and Beckmann (*ibid.*, 1896, 35: 263).

### 10. Paraffin in Confectionery.—Provisional.

Add to the ether extract in the flask, as above obtained, 10 cc of 95 per cent alcohol and 2 cc of 1:1 sodium hydroxid solution, connect the flask with a reflux condenser, and heat for an hour on the water bath, or until saponification is complete. Remove the condenser and allow the flask to remain on the bath until the alcohol is evaporated and a dry residue is left. Treat the residue with about 40 cc of water and heat on the bath, with frequent shaking, until all soluble matter is dissolved. Wash into a separatory funnel, cool, and extract with four successive portions of petroleum ether, which are collected in a tared flask or capsule. Remove the petroleum ether by evaporation and dry in the oven to constant weight.

It should be noted that any phytosterol or cholesterol present in the fat would be extracted with the paraffin, but the amount would be so insignificant that except in the most exacting work it may be disregarded. The character of the final residue should, however, be confirmed by determining its melting point and specific gravity and by subjecting it to examination in the butyrorefractometer.

### 11. Alcohol in Sirups used in Confectionery ("Brandy Drops").<sup>a</sup>—Provisional.

Open each drop by cutting off a section with a sharp knife and collect in a beaker the sirup of from 15 to 25 of the drops, which will usually yield from 30 to 50 grams of sirup. Strain the sirup into a tared beaker through a perforated porcelain plate in a funnel to separate from particles of the inclosing shell, and ascertain the weight of the sirup. Dilute with half its volume of water and determine alcohol as directed under "XIII. Wine," page 83.

### 12. Lead Subacetate Precipitate in Maple Products (Hortvet's Method <sup>b</sup>).—Provisional.

This method consists in reducing the precipitate to a compact mass at the bottom of a graduated tube by the use of a centrifugal machine, noting the reading on the tube, applying necessary corrections, and expressing the volume of the precipitate in cubic centimeters and tenths.

#### (a) APPARATUS.

The apparatus consists of a glass tube and holder, as shown in the illustration, the dimensions being as follows:

Glass tube:	Cm.
Total length.....	15.2
Diameter (wide part).....	3.0
Diameter (neck) .....	2.0
Stem, graduated to 5 cc and tenths, 5 cc division line 5 mm below beginning of wide part.	
Ground area on the wide part for use in numbering.	

<sup>a</sup> Thirty-second Ann. Rept. Mass. Board of Health, 1900, p. 657; reprint, p. 41.

<sup>b</sup> U. S. Dept. Agr., Bureau of Chemistry, Circular 23; J. Amer. Chem. Soc., 1904, 26; 1523.



Wooden holder:

Length	7.7
Diameter	3.2 to 3.5
Diameter of center hole to fit stem	1.3

The tube and holder weigh about 50 grams, and should be so constructed that when fitted together the bottom of the tube will be exactly even with the lower surface of the holder. In a set, each couple, tube and holder, should be made to balance each other. There should be, as nearly as possible, a balanced load carried at the circumference of the wheel of the centrifuge.

## (b) DETERMINATION.

Introduce into the tube 5 cc of sirup or 5 grams of sugar, add 10 cc of water, and dissolve. Add 0.5 cc (10 drops) of alumina cream (prepared as directed under "VI. General Methods," p. 40) and 1.5 cc of lead subacetate and shake thoroughly. Allow the mixture to stand from forty-five to sixty minutes, occasionally giving the tube a twisting motion to facilitate the settling of the precipitate. Place the tube with its holder in the centrifugal machine and run six minutes under the conditions given below. If any material adheres to the sides of the wider portion, remove it by means of a small wire provided with a loop at the end. Return the tube to the centrifuge and run six minutes longer at the same rate. Note the volume of the precipitate, estimating to 0.01 cc as closely as possible. Run a blank, using water and the reagents named above and correct for same. In the case of a sirup the result is reduced to the 5-gram basis by dividing by the specific gravity of the sample. If the sugar content of the sample is known, the specific gravity is found from Table VI, page 221.

The centrifuge used in this method has a radius of 18.5 cm and is run at a speed of 1,600 revolutions per minute. The velocity at the circumference of the wheel is computed in centimeters per second. Calling  $M$  (mass) unity in the formula

$F = \frac{Mv^2}{r}$ , the numerical expression for  $F$ , the centrifugal force, becomes 519,363.

By measuring the radius ( $r$ ) for any given machine and substituting for  $F$ , the numerical constant determined above, the velocity for a given machine may be determined by the following formula,  $v = \sqrt{Fr}$ . Given the velocity in centimeters per second, the required number of revolutions per second or per minute can be computed.

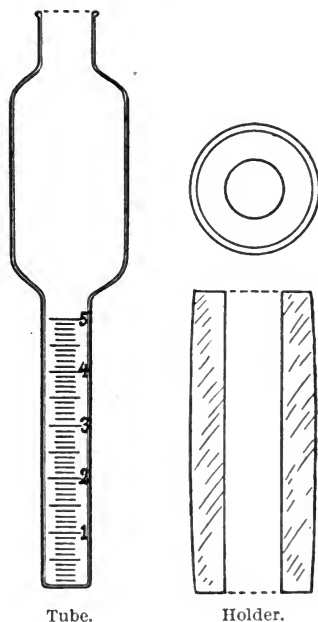


FIG. 1.—Apparatus for the determination of lead subacetate precipitate in maple products.

**13. Malic Acid Value of Maple Products.—Provisional.**

Weigh 6.7 grams of the sample into a 200 cc beaker and add water to make a volume of 20 cc. Add 2 drops of ammonium hydroxid (specific gravity, 0.90), 1 cc of a 10 per cent solution of calcium chlorid, and 60 cc of 95 per cent alcohol. Cover the beaker with a watch glass, heat for one-half hour on a water bath, then turn off the flame and allow the beaker to stand overnight. Filter the material in the beaker through good quality filter paper, wash the precipitate with hot 75 per cent alcohol until the filtrate measures 100 cc, dry and ignite. Add from 15 to 20 cc of tenth-normal hydrochloric acid to the ignited residue, thoroughly dissolve the lime by heating carefully to just below boiling, cool and titrate the excess of acid with tenth-normal sodium hydroxid, using methyl orange as an indicator. One-tenth of the number of cubic centimeters of acid neutralized by the ignited residue expresses the malic acid value. Run blank determinations on reagents, using the same amounts, particularly of ammonium hydroxid, as were used in the original determination, and make the necessary correction.

**14. Detection of Coloring Matter.—Provisional.**

Proceed as directed under "XXVIII, Coloring Matter," page 190.

## XI. METHODS FOR THE ANALYSIS OF SUGARHOUSE PRODUCTS.— PROVISIONAL.

### 1. Sucrose.

#### (a) SCHEIBLER'S ALCOHOLIC METHOD.

In the direct analysis of the beet with the Soxhlet-Sickel apparatus by Scheibler's method, proceed as follows for the extraction of the sucrose:

Place a plug of absorbent cotton in the bottom of the tube, then introduce 26.048 grams (Mohr cc) of the pulped beet, or 32.58 grams (twice the normal weight), according to the polariscope in use, pressing the pulp lightly with a rod. Very small fragments of the beet may be used instead of pulp. Connect the extractor with the reflux condenser. Place 75 cc of 95 per cent alcohol in the flask and connect with the extractor; heat the flask in the water bath and continue the extraction from half an hour to two hours or more, according to the state of division of the sample. Use somewhat weaker alcohol if only 16.29 grams of pulp be taken. Cool and remove the flask, substitute a second containing 75 cc of 75 to 80 per cent alcohol, and continue the extraction to ascertain whether the first extraction were complete.

Fill the first flask to the 100 cc mark, after treating the sample with two or three drops of lead subacetate. Mix the contents of the flask, filter, and polarize. Having extracted the normal weight of pulp, the polariscopic reading is the per cent of sucrose in the sample. The extract in the second flask should also be polarized as a check upon the extraction.

Great care is essential in the polarization of alcoholic solutions. The smallest quantity of lead subacetate that will clarify the solution should be used. The solution must be protected from evaporation during the filtration by a cover glass. Avoid irregularities in the temperature of the solution in the observation tube due to the warmth of the hands, since the density of the solution in different parts of the tube will vary under such conditions; striæ will form, rendering an accurate reading impossible. The Soxhlet extraction apparatus is much more effective than Scheibler's original instrument.

#### (b) PELLET'S AQUEOUS METHOD (HOT DIGESTION).

Any good rasp may be used in the preparation of the pulp for this method. Special flasks with enlarged necks are convenient. Transfer 26.048 grams (Mohr cc) of the pulp to the flask, using a little water to wash the weighing capsule and funnel. The flasks are graduated to contain 200.6 cc for the Schmidt and Haensch, or any instrument using the above normal weight, in

order to compensate for the volume of the marc and the lead precipitate. Add from 3 to 5 cc of lead subacetate solution of 54.3° Brix for the clarification. This reagent should be run into the flask in advance of the beet pulp. Add a few drops of ether to break the foam, then sufficient water to increase the volume of the solution to about 190 cc. Heat to 80° C. in a water bath and maintain this temperature about thirty minutes, occasionally giving the flask a circular movement to facilitate the escape of the air from the pulp. Increase the volume of the solution from time to time during the heating, so that when the operation is completed only a few drops of water will be required to complete the volume of the solution to the mark. After approximately thirty minutes' heating, cool the flask and contents, acidify with strong acetic acid, dilute to the mark, mix, and filter. The state of division of the pulp will govern the time of heating. In polarizing the filtrate use a 400 mm observation tube, thus directly obtaining the per cent of sucrose in the beet.

## **XII. METHODS FOR THE ANALYSIS OF FRUITS AND FRUIT PRODUCTS.**

### **1. Preparation of Sample.—Provisional.**

#### **(a) JUICES, JELLIES, AND SIRUPS.**

Prepare the fresh juices by pressing the well-pulped fruit in a jelly bag and filtering through muslin. In the case of fresh-fruit juices and fresh fruits the determination of total and volatile acids and sugars should be made at once, as fermentation takes place in a very short time. Portions for sucrose and reducing sugar may be weighed and an excess of lead subacetate added. They can then be kept for several days, if desired, without fermentation. All samples must be transferred without delay to glass-stoppered bottles and kept in a cool place.

In the case of jellies, thoroughly mix to insure uniformity in sampling. Weigh 60 grams into a 300 cc flask, dissolve by frequent shaking, make up to the mark with water, and use aliquots for the various determinations. If the jellies contain starch or other insoluble material, thoroughly mix them before taking the aliquots.

#### **(b) FRESH FRUITS.**

Pulp the whole well-cleaned fruit in a large mortar or by means of a food chopper and mix thoroughly. In the case of stone fruits, remove the pits and determine their proportion in a weighed sample.

#### **(c) JAMS, MARMALADES, PRESERVES, AND CANNED FRUITS.**

Thoroughly pulp the entire contents of the jar or can, as directed under fresh fruits; with stone fruits remove the pits and if desired determine their proportion in a weighed sample. In the examination of canned fruits it is often sufficient to merely examine the sirups in which the fruits are preserved. In such cases the liquor may be separated and treated as is prescribed for juices. The relative weights of liquor and fruit may be of value in detecting the presence of an excessive amount of water.

### **2. Alcohol.—Provisional.**

Determine alcohol in 50 grams of the original material according to the method given under "XIII. Wine," page 83.

### **3. Total Solids.—Provisional.**

#### **(a) IN JUICES, JELLIES, AND SIRUPS.**

##### **(1) BY DIRECT DETERMINATION.**

Measure 25 cc<sup>a</sup> of a 20 per cent solution (see 1. (a)) of jelly, or weigh 25 grams of juice into a large flat-bottomed dish which contains about 4 or 5

<sup>a</sup> If a pipette is used it must be graduated so as to deliver a definite volume of a 20 per cent sugar solution after draining a definite time.

grams of freshly ignited asbestos to absorb it; dry for from twenty to twenty-four hours in a water-jacketed oven. In case of jellies that contain starch or insoluble matter, solids may be determined as directed below under (b).

(2) BY CALCULATION FROM SPECIFIC GRAVITY.

Determine the specific gravity of the solution of jelly or diluted sirup, or of the juice as directed under "X. Saccharine Products," page 65.

(b) IN FRESH AND DRIED FRUITS, JAMS, MARMALADES, PRESERVES, AND CANNED GOODS.

Weigh about 20 grams of pulped fresh fruit, or such an amount of fruit products as will give not more than 3 or 4 grams of dried material; add a few cubic centimeters of water, mix thoroughly, and dry as in (a) (1) above.

4. Insoluble Solids.—Provisional.

(a) KREMLA'S METHOD, MODIFIED.

Transfer 50 grams of the sample, by the aid of warm water, to a mortar and thoroughly macerate; then transfer to a muslin filter and wash thoroughly with warm water, care being taken at each addition of water to stir the pulp thoroughly. Collect the filtrate in a 500 cc flask, cool, and make up to volume. Usually this amount is sufficient to remove all soluble material. In extreme cases increase the washings to 1,000 cc; transfer the insoluble residue to an evaporating dish, dry, and weigh.

(b) GERMAN OFFICIAL METHOD.

Transfer a weighed portion of the fruit product to a graduated flask, add water, shake thoroughly, and make up to volume. Allow to settle and either filter or decant off the supernatant liquid. Determine the soluble solids in an aliquot. Total solids less soluble solids equals insoluble solids. The fruit must be thoroughly macerated; the use of a mechanical shaker is advisable.

5. Ash.—Provisional.

(a) DETERMINATION.

Evaporate to dryness 50 cc of the solution of the jelly or diluted sirup (see 1 (a), p. 77), 25 grams of juice or of fresh or canned fruit, or 10 grams of jam, marmalade, preserves, or dried fruit, and determine the ash as directed under "VI. General Methods," page 38.

(b) CONSTITUENTS OF THE ASH.

(1) ALKALINITY.

Into the platinum dish containing the ash run an excess of fifth-normal nitric acid, heat to boiling, cool, and add a few drops of methyl orange.

Carefully rub up the ash with a rubber-tipped stirring rod and titrate the excess of acid with tenth-normal potassium or sodium hydroxid. Calculate the alkalinity to per cent of potassium carbonate in the original substance. One cubic centimeter of tenth-normal acid equals 0.006915 gram of potassium carbonate.

## (2) SULPHATES AND CHLORIDS.

Wash the ash into a 50 cc flask and make up to the mark with water. Evaporate 25 cc of this solution several times to dryness with concentrated hydrochloric acid and determine the sulphates by precipitation with barium chlorid. Multiply the weight of barium sulphate by 0.7469 to obtain the weight of sulphates calculated as potassium sulphate.

In the other portion of the solution determine the chlorin by the Volhard method, as given under "III. Inorganic Plant Constituents," on page 23. The nitric acid added before making the titration will, if it contain enough nitrous oxid, completely destroy the red color of the methyl orange and leave a clear solution for the titration. Calculate the chlorin as per cent of sodium chlorid. Pure fruit jellies and jams give practically no chlorids or sulphates in this amount of ash, but glucose goods may give appreciable amounts. If it is desired to make a complete ash analysis of juices or fresh fruits, much larger amounts must be ashed.

## 6. Total Acidity.—Provisional.

Dilute 25 cc of the solution of jelly or diluted sirup (see (a), p. 77), or 10 grams of juice or fresh fruit, with recently boiled distilled water to about 250 cc, or less if the sample be not highly colored; add phenolphthalein and titrate the acid with tenth-normal alkali. In case of highly colored products litmus paper may be used instead of phenolphthalein. Calculate the results as sulphuric acid.

## 7. Volatile Acids.—Provisional.

Dissolve 25 grams of substance, dilute to 50 cc, and distil in a current of steam, as directed under "XIII. Wines," page 86, section 13. Each cubic centimeter of tenth-normal alkali is equivalent to 0.006 gram of acetic acid.

## 8. Detection of Free Mineral Acids.—Provisional.

Use Hehner's method, as given under "XVI. Vinegar," page 104.

## 9. Nitrogen.—Provisional.

Use 5 grams of jelly or other fruit product, or 10 grams of juice or fresh fruit, for the determination of nitrogen according to either the Gunning or the Kjeldahl method. ("I. Fertilizers," p. 5.) Express results as protein (nitrogen multiplied by 6.25).

## 10. Sucrose.—Provisional.

## (a) BY POLARIZATION.

Determine by polarizing before and after inversion, as directed on page 40, under "VI. General Methods."

## (b) BY REDUCTION.

Determine as directed on page 41, under "VI. General Methods."

## 11. Reducing Sugars.—Provisional.

Determine as directed on page 42, under "VI. General Methods."

**12. Dextrin.—Provisional.**

Dissolve 10 grams of the sample in a 100 cc flask, add 20 mg of potassium fluorid, and then about one-quarter of a cake of compressed yeast. Allow the fermentation to proceed below 25° C. for two or three hours to prevent excessive foaming, and then place in an incubator at a temperature of from 27° to 30° C. for five days. At the end of that time, clarify with lead subacetate and alumina cream, make up to 100 cc, and polarize in a 200 mm tube. A pure fruit jelly will show a rotation of not more than a few tenths of a degree either to the right or to the left. If a polariscope having the Ventzke scale be used and a 10 per cent solution be polarized in a 200 mm tube, the number of degrees read on the sugar scale of the instrument multiplied by 0.8755 will give the percentage of dextrin, or the following formula may be used:

$$\text{Percentage of dextrin} = \frac{C \times 100}{198 \times L \times W}$$

In which

C=degrees of circular rotation.

L=length of tube in decimeters.

W=weight of sample in 1 cubic centimeter.

**13. Alcohol Precipitate.—Provisional.**

Evaporate 100 cc of a 20 per cent solution of jelly (see 1 (a), p. 77), diluted sirup, or of the washings from the determination of insoluble solids, to 20 cc; add slowly and with constant stirring 200 cc of 95 per cent alcohol and allow the mixture to stand over night. Filter and wash with 80 per cent alcohol by volume. Wash this precipitate off the filter paper with hot water into a platinum dish; evaporate to dryness; dry at 100° C. for several hours and weigh; then burn off the organic matter and weigh the residue as ash. The loss in weight upon ignition is called alcohol precipitate.

The ash should be largely lime and not more than 5 per cent of the total weight of the alcohol precipitate. If it is larger than this some of the salts of the organic acids have been brought down. Titrate the water-soluble portion of this ash with tenth-normal acid, as any potassium bitartrate precipitated by the alcohol can thus be estimated.

The general appearance of the alcohol precipitate is one of the best indications as to the presence of glucose and dextrin. Upon the addition of alcohol to a pure fruit product a flocculent precipitate is formed with no turbidity, while in the presence of glucose a white turbidity appears at once upon adding the alcohol, and a thick, gummy precipitate forms.

**14. Tartaric, Citric, and Malic Acids (Schmidt-Hiepe Method Modified).—Official.**

Use the filtrate from the alcohol precipitate in this determination. After evaporating the alcohol and taking up the acids with water add lead subacetate until the solution is alkaline, then filter and wash the precipitate until only a slight amount of lead remains in the washings. Wash the precipitate off the filter paper into a beaker with hot water, precipitate the lead by hydrogen sulphid, and filter off the lead sulphid while hot, washing with hot water. Evaporate the filtrate which contains the free organic acids to about 50 cc, neutralize with potassium hydroxid, add an excess of strong solution of neutral



calcium acetate with constant stirring, and allow to stand from six to twelve hours. Throw the precipitate of calcium tartrate on a filter paper and wash until filtrate and washings make exactly 100 cc; ignite the filter paper and precipitate, and determine the lime by titration. A correction of 0.0286 gram of tartaric acid, which is held in solution in the 100 cc of washings as calcium tartrate, must be added. Evaporate the filtrate down to about 20 cc, and if a precipitate of calcium citrate is formed collect it on a filter while hot, wash with hot water, ignite, and titrate. From this result calculate the citric acid. Evaporate the filtrate and washings from the calcium citrate to about 20 cc and add three volumes of 96 per cent alcohol by volume, which will throw down the calcium salt of tartaric acid held in solution, the remaining citrate, and the malate and succinate. Filter, ignite the precipitate, titrate, and calculate as malic acid after subtracting the tartaric acid present. (The amount of citric and succinic acid present is very small.)

#### 15. Tartaric Acid.—Provisional.

Determine tartaric acid in 100 cc of the fruit juice as directed for total tartaric acid under "XIII. Wine," page 86, except that 20 cc of alcohol is used in the precipitation instead of 15 cc.

#### 16. Citric Acid.<sup>a</sup>—Provisional.

Evaporate 50 cc of the fruit solution on a water bath to a sirupy condition. To the residue add, very slowly at first and with constant stirring, 95 per cent alcohol until no further precipitate is formed, 70 to 80 cc are generally enough. Filter and wash the residue with 95 per cent alcohol. Evaporate the filtrate to eliminate the alcohol, take up the residue with a little water and transfer to a graduated cylinder, making up to 10 cc. To 5 cc of this solution add half a cubic centimeter of glacial acetic acid, and, drop by drop, a saturated solution of lead acetate. The presence of citric acid is shown by the appearance of a precipitate which dissolves when heated and reappears when cooled. In order to separate the citric acid from other acids, heat to boiling, filter, and wash with boiling water, then allow to cool and the precipitate of lead citrate will again form. This lead precipitate may be filtered off, washed with weak alcohol, dried, weighed, and the citric acid calculated. It is necessary that no tartaric acid be present. If the tartaric acid has been estimated, any error on this account may be avoided by adding enough tenth-normal potash to neutralize the tartaric acid before the alcohol is added.

#### 17. Detection of Preservatives.—Provisional.

Test as directed on page 179, under "XXVII. Food Preservatives."

#### 18. Detection of Coloring Matter.—Provisional.

Follow the directions given under "XXVIII. Coloring Matter," page 190.

#### 19. Detection of Artificial Sweetening Material.—Provisional.

Follow the directions given under "XXVII. Food Preservatives," pages 182 and 189.

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<sup>a</sup> Möslinger, Zts. Nahr. Genussm., 1899, 2: 93.

**20. Detection of Starch.—Provisional.**

First destroy the color of the jelly by treatment with sulphuric acid and potassium permanganate and then test with iodine. Bring the solution of jelly nearly to the point of boiling, add several cubic centimeters of dilute sulphuric acid and then potassium permanganate until all color is destroyed. The starch remains unaffected by this treatment. The presence of starch is not necessarily an indication of its addition as an adulterant. It is usually present in small amounts in the apple, and occasionally in other fruits, and unless it is found in the fruit product in considerable amounts its presence may be due to these natural sources.

**21. Detection of Gelatin.<sup>a</sup>—Provisional.**

The presence of gelatin in jellies and jams is shown by the increased content of nitrogen. Precipitate a concentrated solution of jelly or jam with 10 volumes of absolute alcohol and determine nitrogen in the dried precipitate by the Kjeldahl or Gunning method. ("I. Fertilizers," p. 5.)

**22. Detection of Agar Agar.<sup>b</sup>—Provisional.**

Heat the jelly with 5 per cent sulphuric acid, add a crystal of potassium permanganate, and allow to settle. If agar agar is present the sediment will be rich in diatoms, which can be detected by the use of the microscope.

**23. Heavy Metals.—Provisional.**

Proceed as directed under "IX. Canned Vegetables," page 61.

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<sup>a</sup> A. Boemer, Chem. Ztg., 1895, 19: 552.

<sup>b</sup> G. Marpmann, Zts. angew. Mikrosk., 1896, 2: 260.

### **XIII. METHODS FOR THE ANALYSIS OF WINE.**

The determinations of most value in judging the purity of wine are alcohol, glycerol, extract, ash, total and volatile acids, and reducing sugar. The actual percentage of these substances present is of interest, but much more important are certain relations between them, such as ash to extract, extract to alcohol, alcohol to glycerol, alcohol to acids, and volatile acids to total acids. Examination for preservatives and foreign coloring matter must also be made. In the examination of sparkling wines, remove carbon dioxide by shaking.

#### **1. Specific Gravity.—Provisional.**

Determine at 15.6° C. by means of a pycnometer, a small accurately graduated hydrometer, or a Westphal plummet on the analytical balance. If a pycnometer be used it should be warmed quickly to room temperature after filling and before weighing, to prevent the error due to the collection of moisture on the outside. A small hole filed in the cap will permit the necessary expansion in the volume of the liquid.

#### **2. Alcohol.—Provisional.**

Measure 100 cc of the liquid at 15.6° C. into a distilling flask of from 250 to 300 cc capacity; add 50 cc of water; attach the flask to a vertical condenser by means of a bent tube and distil almost 100 cc, making up to 100 cc volume when cooled to 15.6° C. Foaming, which sometimes occurs, especially with new wines, may be prevented by the addition of a small amount of tannin. If it be desired to determine the alcohol in wines which have undergone acetic fermentation and contain a large amount of acetic acid, 0.1 or 0.2 gram of precipitated calcium carbonate should be added. This is unnecessary, however, in wines of normal taste and odor. Determine the specific gravity of the distillate as directed under "1. Specific Gravity," and obtain the corresponding percentage of alcohol, by volume and grams per 100 cc, from Table II, page 203. Multiply the per cent of alcohol by the weight of the distillate (corresponding to the specific gravity in Table II) and divide the result by the weight of the sample (calculated, from the specific gravity) to obtain the per cent of alcohol by weight.

#### **3. Glycerol.—Provisional.**

##### **(a) IN DRY WINES.**

Evaporate 100 cc of wine in a porcelain dish on the water bath to a volume of about 10 cc and treat the residue with about 5 grams of fine sand and with from 3 to 4 cc of milk of lime (containing about 15 per cent of calcium oxid) for each gram of extract present, and evaporate almost to dryness. Treat the moist residue with 50 cc of 90 per cent alcohol by volume, remove the substance adhering to the sides of the dish with a spatula, and rub the whole mass to a paste. Heat the mixture on the water bath, with constant stirring, to incipient

boiling and decant the liquid through a filter into a small flask. Wash the residue repeatedly by decantation with 10 cc portions of hot 90 per cent alcohol until the filtrate amounts to about 150 cc. Evaporate the filtrate to a sirupy consistency in a porcelain dish, on a hot, but not boiling, water bath; transfer the residue to a small glass-stoppered graduated cylinder with 20 cc of absolute alcohol, and add 3 portions of 10 cc each of absolute ether, thoroughly shaking after each addition. Let stand until clear, then pour off through a filter, and wash the cylinder and filter with a mixture of one part of absolute alcohol to one and one-half parts of absolute ether, pouring the wash liquor also through the filter. Evaporate the filtrate to a sirupy consistency, dry for one hour at the temperature of boiling water, weigh, ignite, and weigh again. The loss on ignition gives the weight of glycerol.

#### (b) IN SWEET WINES.

With wines whose extract exceeds 5 grams per 100 cc, heat to boiling in a flask the portion to be used in the determination of glycerol, and treat with successive small portions of milk of lime until it becomes first darker and then lighter in color. When cool add 200 cc of 95 per cent alcohol, allow the precipitate to subside, filter, and wash with 95 per cent alcohol. With the filtrates thus obtained proceed as directed under (a).

#### 4. Glycerol-Alcohol Ratio.—Provisional.

Express this ratio as  $x:100$ , in which  $x$  is obtained by multiplying the percentage by weight of glycerol by 100 and dividing the result by the percentage by weight of alcohol.

#### 5. Extract.—Provisional.

##### (a) FROM SPECIFIC GRAVITY OF DEALCOHOLIZED WINE.

Preliminary to its exact determination the extract should be calculated by the formula:

$$sp.=1+a-b$$

In which "sp." is the specific gravity of the dealcoholized wine, "a" the specific gravity of the wine, and "b" the specific gravity of the alcoholic distillate obtained in the estimation of alcohol.

Illustration: A sample of Catawba is examined with the following result:

Specific gravity of wine (a)-----	1.0402
Specific gravity of alcoholic distillate (b)-----	.9857
Difference (a-b)-----	.0545
Specific gravity of dealcoholized wine (1+a-b)-----	1.0545
Extract (grams per 100 cc)-----	14.12

The extract equivalent of "sp." is obtained from Table V, page 218.

#### (b) BY EVAPORATION.

##### (1) IN DRY WINES.

(Having an extract content of less than 3 grams per 100 cc.)

Evaporate 50 cc of the sample on the water bath to a sirupy consistency in a flat-bottom platinum dish approximately 85 mm in diameter and of about 75 cc capacity. Heat the residue for two and one-half hours in a drying oven at the temperature of boiling water and weigh. The sugar-free extract is

found by deducting the weight of sugar in excess of 0.1 gram per 100 cc from the total residue. In the case of plastered wines, the potassium sulphate in excess of 0.1 gram is also deducted.

## (2) IN SWEET WINES.

When the extract content is between 3 and 6 grams per 100 cc treat 25 cc of the sample as described under dry wines. When the extract exceeds 6 grams per 100 cc, however, the result obtained under (a) is accepted, and no gravimetric determination is attempted. This is because of the serious error connected with drying levulose at high temperature. (The table referred to under (a) was obtained by drying at 75° C. in vacuo.)

### 6. Ash.—Provisional.

Proceed as directed under "VI. General Methods," on page 38, employing the residue from the determination of the extract.

### 7. Ash-Extract Ratio.—Provisional.

Express results as 1:x, in which x is the quotient obtained by dividing the percentage of extract by the percentage of ash.

### 8. Sodium Chlorid.—Provisional.

Calculate from the chlorin in the ash, determined as directed on page 23, under "III. Inorganic Plant Constituents."

### 9. Potassium Sulphate.—Provisional.

Precipitate sulphuric acid directly in 50 cc of wine by means of barium chlorid after acidifying with hydrochloric acid and determine the resulting barium sulphate by the ordinary method.

### 10. Phosphoric Acid.—Provisional.

Determine phosphoric acid in the ash by one of the official methods given under "III. Inorganic Plant Constituents," page 22.

### 11. Barium and Strontium.<sup>a</sup>—Provisional.

Evaporate to dryness 100 cc of wine, incinerate as directed under the determination of ash "VI. General Methods," page 38, dissolve in dilute hydrochloric acid, evaporate to dryness, and examine the residue spectroscopically. If barium or strontium be present, fuse with sodium carbonate to decompose silicates,<sup>b</sup> and determine by precipitation with sulphuric acid.

### 12. Total Acids.—Provisional.

Transfer 25 cc of the sample to a beaker, heat to incipient boiling, and in the case of white wines add about 10 drops of a neutral litmus solution and titrate while still hot with tenth-normal sodium hydroxid. With red wines add sodium hydroxid solution until the red color changes to violet and continue to add a few drops at a time until a drop of the mixture placed on delicate red litmus

<sup>a</sup> Borgmann, *Analyse des Weines*, 2d ed., page 143.

<sup>b</sup> R. Fresenius, *Zts. anal. Chem.*, 1890, 29: 20, 143 and 413; 1891, 30: 18, 452 and 583; 1893, 32: 189 and 312.

paper shows an alkaline reaction. The result is expressed in terms of tartaric acid. One cubic centimeter of tenth-normal sodium hydroxid is equivalent to 0.0075 gram tartaric acid.

### 13. Volatile Acids.—Provisional.

Distil in a current of steam 50 cc of wine, to which a little tannin has been added to prevent foaming. Heat the flask containing the sample until the liquid boils, regulate the flame under it so that the volume remains constant, and pass the steam through until 200 cc have been collected in the receiver. Titrate the distillate with tenth-normal sodium hydroxid, using phenolphthalein as indicator and express the result as acetic acid.

One cubic centimeter of tenth-normal sodium hydroxid is equivalent to 0.006 gram acetic acid.

### 14. Fixed Acids.—Provisional.

The amount of fixed acids is ascertained by subtracting 1.25 times the volatile acids from the total acids expressed as tartaric.

### 15. Tartaric Acid and Tartrates.—Provisional.

#### (a) TOTAL TARTARIC ACID.<sup>a</sup>

To 100 cc of wine add 2 cc of glacial acetic acid, 3 drops of a 20 per cent solution of potassium acetate, and 15 grams of powdered potassium chlorid, and stir to hasten solution. Add 15 cc of 95 per cent alcohol and rub the side of the beaker vigorously with a glass rod for about one minute to start crystallization. Let stand at least fifteen hours at room temperature; decant the liquid from the separated acid potassium tartrate as rapidly as possible on a gooch prepared with a very thin film of asbestos, transferring no more of the precipitate to the crucible than necessary. Wash the precipitate and filter three times with a small amount of a mixture of 15 grams of potassium chlorid, 20 cc of 95 per cent alcohol, and 100 cc of water, using not more than 20 cc of the wash solution in all. Transfer the asbestos film and precipitate to the beaker in which the precipitation took place, wash out the gooch with hot water, add about 50 cc of hot water, heat to boiling, and titrate the hot solution with tenth-normal sodium hydroxid, using delicate litmus tincture or litmus paper as indicator. Increase the number of cubic centimeters of tenth-normal alkali employed by 1.5 cc, on account of the solubility of the precipitate. One cubic centimeter of tenth-normal alkali so consumed is equivalent to 0.0150 gram of tartaric acid.

#### (b) CREAM OF TARTAR.—PROVISIONAL.

Determine the alkalinity of the soluble ash as directed under "XVI. Vinegar," page 102. One cubic centimeter of tenth-normal alkali is equivalent to 0.0188 gram of potassium bitartrate.

#### (c) FREE TARTARIC ACID.—PROVISIONAL.

Add 25 cc of tenth-normal hydrochloric acid to the ash of 50 cc wine, heat to incipient boiling, and titrate with tenth-normal sodium or potassium hydroxid, using litmus as indicator. Deduct the number of cubic centimeters of alkali employed from 25 and multiply the remainder by 0.0075 to obtain the

<sup>a</sup> Halenke and Möslinger, *Zts. anal. Chem.*, 1895, **34**: 263.

amount of tartaric acid necessary to combine with all the ash (considering it to consist entirely of potash). Deduct the figure so obtained from the total tartaric acid for the free tartaric acid.

#### 16. Crude Protein.—Provisional.

Determine nitrogen in 50 cc of wine by the Kjeldahl or Gunning method (see page 5, under "I. Fertilizers") and multiply the result so obtained by 6.25.

#### 17. Sugar.

##### (a) BY REDUCTION.—PROVISIONAL.

Place 200 cc of wine in a porcelain dish, exactly neutralize with an approximately normal sodium hydroxid, using litmus paper as indicator, and evaporate to about one-fourth the original volume. Transfer to a 200 cc flask, add sufficient normal lead acetate to clarify, dilute to the mark with water, shake, and filter through a folded filter. Remove the lead and determine reducing sugars before and after inversion by the Soxhlet method, page 42, under "VI. General Methods."

##### (b) BY POLARIZATION.—PROVISIONAL.

Polarize part of the filtrate obtained in (a), before and after inversion, in a 200 mm tube as directed under "VI. General Methods," page 40, to obtain the polarization of the wine in the original dilution. In calculating the percentage of sucrose the relation of the amount of sample to the normal weight must be taken into consideration.

#### 18. Commercial Glucose.—Provisional.

Wine polarizing over  $0.9^{\circ}$  to the right and containing not more than 0.1 per cent of reducing sugar may have been prepared from glucose, and should be treated as follows:

Dealcoholize 200 cc of wine by evaporating to about one-fourth its volume, and add enough water to the residue to make its sugar content less than 15 per cent. For the purpose of this operation the sugar content of the wine may be assumed to be 2 per cent less than the extract. Add 2 or 3 grams of compressed yeast, let stand at about  $25^{\circ}$  C. for four or five days, when fermentation will be complete. Evaporate the fermented liquid in a porcelain dish to a thin sirup, after the addition of a little sand and a few drops of a 20 per cent solution of potassium acetate and acetic acid. To the residue add 200 cc of 90 per cent alcohol, with constant stirring. Separate the alcohol solution by filtration and evaporate until about 5 cc remain. Mix the residue with washed boneblack, filter into a graduated cylinder, and wash until the filtrate (cooled to  $15^{\circ}$  C.) amounts to 30 cc. When the filtrate shows a dextrorotation of more than  $1.5^{\circ}$ , it indicates the presence of the unfermentable constituents of commercial glucose. Results by this method are not reliable with wines that are heavily preserved.

#### 19. Gum and Dextrin.—Provisional.

Evaporate 100 cc of wine to about 10 cc and add 10 cc of 96 per cent alcohol. If gum or dextrin be present (indicated by the formation of a voluminous precipitate), continue the addition of alcohol slowly and with stirring until 100 cc have been added. Let stand over night, filter, and wash with 80 per cent alcohol by volume. The precipitate may then be dried and weighed, or it may be treated

according to the modified Sachsse method for the determination of starch. (See "VI. General Methods," p. 53.)

## 20. Tannin and Coloring Matter.—Official.

### (a) PREPARATION OF REAGENTS.

(1) *Oxalic acid*.—Use tenth-normal solution; 10 cc=0.04157 gram of tannin.

(2) *Potassium permanganate solution*.—Dissolve 1.333 grams of potassium permanganate in 1 liter of water and standardize the solution with the tenth-normal oxalic acid solution.

(3) *Indigo solution*.—Dissolve 6 grams of sodium sulphindigotate in 500 cc of water with the aid of heat; cool, add 50 cc of concentrated sulphuric acid, make the solution up to 1 liter, and filter.

(4) *Purified boneblack*.—Extract finely pulverized boneblack with hydrochloric acid and wash with distilled water until the acid is entirely removed. The boneblack is kept covered with water.

### (b) DETERMINATION.<sup>a</sup>

Dealcoholize 100 cc by evaporation and dilute with water to the original volume. Transfer 10 cc to a porcelain dish of about 2 liters capacity; add about a liter of water and exactly 20 cc of indigo solution. Add tenth-normal potassium permanganate solution, a cubic centimeter at a time, until the blue color changes to green; then add a few drops at a time until the color becomes golden yellow. Designate the number of cubic centimeters of permanganate solution employed as "a."

Treat 10 cc of the dealcoholized wine, prepared as above, with boneblack for fifteen minutes; filter and wash the boneblack thoroughly with water. Add a liter of water and 20 cc of indigo solution and titrate with permanganate as above. Designate the number of cubic centimeters of permanganate employed as "b."

Then  $a-b=c$ , the number of cubic centimeters of permanganate solution required for the oxidation of the tannin and coloring matter in 10 cc of wine.

## 21. Heavy Metals.—Provisional.

Lead and copper may be determined in 500 or 1,000 cc by the method given under "IX. Canned Vegetables," page 62.

Copper may also be precipitated electrolytically<sup>b</sup> on 500 cc of the undiluted wine by using as electrodes pieces of platinum foil 3 by 15 cm. Arsenic may be detected or determined by one of the modifications of the Marsh method.

## 22. Detection of Nitrates.

### (a) WHITE WINE.—PROVISIONAL.

Treat a few drops of the wine in a porcelain dish with 2 or 3 cc of concentrated sulphuric acid which contains about 0.1 gram of diphenylamin<sup>c</sup> per

<sup>a</sup> Neubauer-Löwenthal method, *Ann. der Oenologie*, 2: 1.

<sup>b</sup> Fruhauf and Ursic, Bericht über die Verammlung Oesterreichischer Oenomiker in Bozen, 1886, 1: 66; Borgmann, *Analyse des Weines*, 2d ed., p. 146.

<sup>c</sup> Egger, *Arch. Hyg.*, 2: 273.



100 cc. The deep blue color formed in the presence of nitrates appears so quickly that it is not obscured, even in sweet wine, by the blackening produced by the action of sulphuric acid on the sugar.

(b) RED WINE.—PROVISIONAL.

Clarify with lead subacetate and remove the excess of lead with sodium sulphate as directed under the determination of sugar ("VI. General Methods," p. 40). Filter and treat a few drops of the filtrate as directed under (a).

**23. Detection of Foreign Coloring Matter.—Provisional.**

Follow the directions given under "XXVIII. Coloring Matter," page 190.

**24. Detection of Preservatives.**

Follow the methods given under "XXVII. Food Preservatives," page 179 and following.

The detection of added boric acid is somewhat difficult because a small amount of it is normally present in certain wines. To be of practical value, therefore, this test should be quantitative. The determination of sulphurous acid must also be quantitative. A small amount of salicylic acid is also normal in wine, and for that reason not more than 50 cc of the sample should be used in testing for that preservative.

## **XIV. METHODS FOR THE ANALYSIS OF BEER.—PROVISIONAL.**

### **1. Preparation of Sample.**

Transfer the contents of the bottle or bottles to a large flask and shake vigorously to hasten the escape of carbon dioxid, care being taken that the beer is not below 15° C., since below this temperature the carbon dioxid is retained in the beer and is liable to form bubbles in the pycnometer.

### **2. Specific Gravity.**

Determine specific gravity as directed under "XIII. Wine," on page 83.

### **3. Alcohol.**

#### **(a) DISTILLATION METHOD.**

Determine as directed under "XIII. Wine," on page 83.

#### **(b) OPTIONAL METHOD.**

Calculate the alcohol content from the reading of the Zeiss immersion refractometer on the distillate, reporting the results at 20° C.

### **4. Extract.**

#### **(a) METHOD I.**

Evaporate 25 cc of the beer in a tared platinum dish to constant weight in a water oven at 80° C.

#### **(b) OPTIONAL METHOD II.**

Calculate according to formula  $sp = g + (1 - a)$ , in which  $sp$  is the specific gravity of the dealcoholized beer,  $g$  the specific gravity of the beer,  $a$  the specific gravity of the distillate obtained in the determination of alcohol, and determine value of  $sp$  from standard tables on extract in beer wort. (Tables III and IV, pp. 209 and 214.)

#### **(c) OPTIONAL METHOD III.**

Make immersion refractometer reading of dealcoholized beer and calculate extract in grams per 100 cc.

### **5. Extract and Specific Gravity of Original Wort.**

Calculate percentage of extract from the formula  $O = 2A + E$ , when  $O$ =original extract of wort,  $A$ =alcohol by weight, and  $E$ =extract of dealcoholized beer. From extract calculated as above compute from standard tables the specific gravity of the wort. (See Tables III and IV, pp. 209 and 214.)

### 6. Degree of Fermentation.

200 A

Calculate from the formula  $D = \frac{200A}{B}$  in which D is the degree of fermentation.

B

A=percentage of alcohol by weight, and B the original extract.

### 7. Total Acids.

(A) Heat 20 cc of the sample to incipient boiling to liberate carbon dioxide, and titrate with tenth-normal sodium hydroxide, using neutral litmus as indicator. Each cubic centimeter of tenth-normal alkali employed is equivalent to 0.009 gram of lactic acid. The number of cubic centimeters of tenth-normal alkali employed in titrating 20 cc of beer is multiplied by 0.045 for the acidity expressed to grams of lactic acid per 100 cc.

(B) Calculate the cubic centimeters of tenth-normal sodium hydroxide required to neutralize the acidity of 100 cc of the sample.

### 8. Volatile Acids.

(A) The volatile acid, as acetic acid, is determined by titrating 20 cc of the alcoholic distillate with tenth-normal sodium hydroxide solution, using phenolphthalein as an indicator. The number of cubic centimeters of tenth-normal alkali employed multiplied by 0.030 gives the acidity expressed as grams of acetic acid per 100 cc.

(B) Calculate the cubic centimeters of tenth-normal sodium hydroxide required to neutralize the acidity of 100 cc of sample.

### 9. Reducing Sugars.

Dilute 25 cc of beer, free from carbon dioxide, with water to 100 cc. Determine the reducing sugar in 25 cc of this solution as directed on page 42, under "VI. General Methods," the solution being boiled four minutes instead of two. Express the result in terms of maltose equivalent to copper reduced according to the table given on page 47.

### 10. Dextrin.

#### (a) SACHSSE-ALLIHN METHOD.

Dilute 50 cc of beer and 15 cc of hydrochloric acid, specific gravity 1.25, to 200 cc, attach to a reflux condenser, and keep in a boiling-water bath for two hours. Employ Sachsse's method for the hydrolyzation of starch and determine dextrose according to Allihn. ("VI. General Methods," pp. 49 and 53.) The amount of dextrose thus found multiplied by 20 (or 24 if diluted to 300 cc) and divided by the specific gravity equals the dextrose in the original beer. From this figure subtract 95 per cent of the amount of maltose in the original beer and multiply the remainder by 0.9, the result being the percentage of dextrin in the original wort.

#### (b) OPTIONAL METHOD.

Dextrin may be determined by the following method, based on the difference between its optical activity and that of maltose.

$D = \frac{A - (M \times 8.1)}{11.6}$  when D=dextrin, in grams per 100 cc, A=total rotation in degrees Ventzke in 200 mm tube, and M=percentage of maltose as determined gravimetrically.

### 11. Direct Polarization.

Read the polarization of the original sample in degrees Ventzke in a 200 mm tube. If the beer is turbid, clarify by shaking with alumina cream.

### 12. Invert Polarization.

To 10 cc of the beer in a small flask add 1 cc of concentrated hydrochloric acid, invert by slowly heating to 68° C., cool, polarize in a 200 mm tube, and increase the reading one-tenth to allow for dilution.

### 13. Glycerol.

Proceed as directed on page 83 under "XIII. Wine." The milk of lime is added during evaporation after the carbon dioxide has been expelled. It is advisable that the filtrate after being evaporated to a sirupy consistency be treated again with 5 cc of absolute alcohol and 2 portions of 7.5 cc each of absolute ether. If clear, continue as directed. If not clear, it is necessary to repeat the treatment with lime.

### 14. Ash.

Evaporate 25 cc of the sample to dryness, and determine according to method given under "VI. General Methods," page 38.

### 15. Phosphoric Acid.

Measure out 50 cc of the original beer, free from carbonic acid, into a small beaker. Add 5 cc of an acid solution of sodium acetate and heat to boiling. Run in with a burette, standard uranium solution, 0.5 cc at a time, testing each time until a drop of the beer when placed on a white plate colors a drop of potassium ferrocyanide slightly brown. The number of cubic centimeters of the uranium-acetate solution necessary, multiplied by 0.01 gives the grams per 100 cc of phosphoric acid in the beer. If the beer is very dark, employ the official gravimetric or volumetric method, using the residue obtained in the determination of the ash. ("I. Fertilizers," pp. 1 and 4.)

### 16. Protein.

Evaporate 25 cc of the original beer, to which has been added a small amount of tannin to prevent frothing, and proceed according to the Kjeldahl or Gunning method for the determination of nitrogen and multiply the result by 6.25. ("I. Fertilizers," p. 5.)

### 17. Carbon Dioxide.

#### (a) BOTTLED GOODS.

Pierce the cork with a champagne tap.<sup>a</sup> Connect with a suitable absorption apparatus, placing an Erlenmeyer flask between the bottle and absorption tubes

<sup>a</sup> Hassall, Food: Its Adulterations and Methods for their Detection, 1876, p. 668. Used by Wiley (Amer. Chem. J., 1886, 8: 200) in the examination of koumiss, and by Crampton in the examination of beer. Crampton found it necessary to regrind the cocks and ream off the thread, leaving a smooth tube. (U. S. Dept. Agr., Division of Chemistry, Bul. 13, pt. 3, p. 294.)

to allow the bubbles to break and prevent them from passing beyond it. The accompanying illustration (fig. 2) of an apparatus devised by Crampton and Trescott<sup>a</sup> answers admirably for this purpose. Immerse the bottle in water in a suitable vessel—such as an ether can with the top cut away, as shown in the cut—allow the gas to escape slowly, and when it ceases to flow spontaneously heat gradually to about 80° C. and maintain this temperature for about half an hour, shaking the bottle from time to time. Then disconnect the bottle, replace it with a soda-lime tube, and draw a current of air through the apparatus. The increase in weight of the absorption tube gives the amount of carbon dioxide. The beer employed is finally either weighed or measured.

When the bottle is closed with a patent stopper, the latter may sometimes be replaced by a rubber stopper fitted with a stopcock tube. Where the pressure

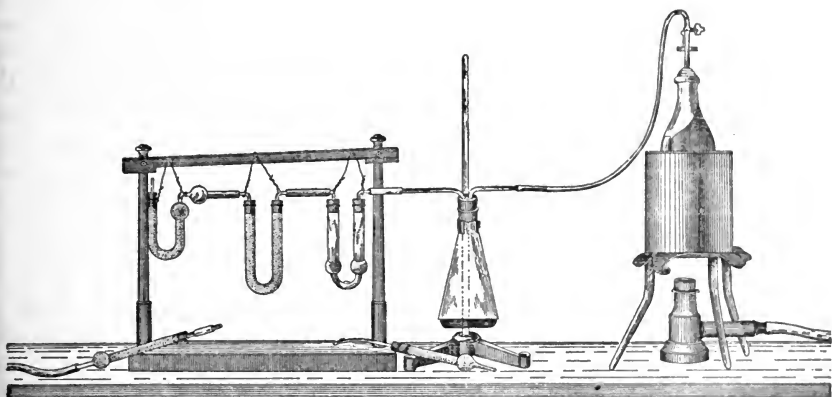


FIG. 2.—Apparatus for the determination of carbon dioxide in beer.

is so great that this is not practicable, such samples may be treated as directed below under "Bulk Goods."

#### (b) BULK GOODS.

Close a round-bottom flask of about 700 cc capacity with a two-hole rubber stopper fitted with two stopcock tubes bent at right angles—one passing to the bottom of the flask and the other ending just below the stopper.<sup>b</sup> Produce a partial vacuum in the flask by means of an aspirator, and weigh the flask. Dip the end of one of the stopcock tubes below the surface of the beer, or, better, attach it by means of a rubber tube to a champagne tap or small faucet screwed into the cask, and allow about 300 cc of the sample to enter the flask. Weigh the flask and contents, and proceed as directed under "Bottled Goods." Somewhat better results may be obtained by placing a reflux condenser between the flask and absorption apparatus, and heating the flask over a burner to the boiling point. Attach the stopcock tube to a soda-lime guard tube and pass a current of air through the apparatus. The amount of carbon dioxide is ascertained by the increase in weight of the absorption tube.

<sup>a</sup> U. S. Dept. Agr., Division of Chemistry, Bul. 13, pt. 3, p. 293.

<sup>b</sup> Windisch (*Das chemische Laboratorium des Brauers*, p. 247) employs ordinary glass tubes provided with rubber tubing and screw cocks.

**18. Preservatives.**

Add 5 cc of dilute sulphuric acid to 100 cc of beer from which the alcohol has been driven off, and determine as directed under "XXVII. Food Preservatives," page 179.

**(a) SULPHITES AND SULPHUROUS ACID.**

Determine in 50 cc of the beer as directed under "XXVII. Food Preservatives," page 187.

**(b) FLUORIDS—METHOD OF BLAREZ.**

Thoroughly mix the sample and heat 150 cc to boiling. Add to the boiling liquid 5 cc of a 10 per cent solution of barium acetate. Collect the precipitate in a compact mass, using to advantage a centrifuge, wash upon a small filter and dry in the oven. Transfer to a platinum crucible, first breaking up the dry precipitate and then adding the filter ash to the crucible, and complete the determination, as directed under "XXVII. Food Preservatives," page 186.

## **XV. METHODS FOR THE ANALYSIS OF DISTILLED LIQUORS.**

### **1. Specific Gravity.—Provisional.**

Determine at 15.6° C. as described on page 83, under "XIII. Wine."

### **2. Alcohol by Weight.—Official.**

Weigh into a distilling flask 20-25 grams of the sample, dilute with 100 cc of water, distil nearly 100 cc, and either weigh distillate or make to volume at 15.6° C, and determine the specific gravity. Obtain the percentage of alcohol by weight corresponding to the specific gravity from Table II (p. 203); multiply this figure by the weight of the distillate and divide by the weight of the sample taken to obtain the per cent by weight of alcohol.

### **3. Alcohol by Volume.—Official.**

From the specific gravity of the distillate obtained under "2" find the percentage of alcohol by volume from Table II (p. 203). Multiply this figure by the volume of distillate (calculated from the specific gravity) and divide by the volume of the sample, thus obtaining the percentage of alcohol by volume in the original sample.

### **4. Extract.—Official.**

Weigh or measure (at 15.6° C.) 100 cc of the sample, evaporate nearly to dryness on the water bath, then transfer to a water oven, and dry at the temperature of boiling water for two and one-half hours.

### **5. Ash.—Provisional.**

Proceed as directed under "VI. General Methods" (p. 38), employing the residue from the determination of the extract (4).

### **6. Acidity.—Provisional.**

Titrate 100 cc (or 50 cc diluted to 100 cc if the sample is dark in color) with tenth-normal alkali, using phenolphthalein as indicator. One cubic centimeter of tenth-normal alkali is equal to 0.006 of acetic acid.

### **7. Ethereal Salts.—Provisional.**

Use 50 cc of the distillate prepared as follows: Add 25 cc of water to 200 cc of the sample and distil slowly 200 cc, using a mercury valve to prevent loss of alcohol. Exactly neutralize the free acid with tenth-normal alkali, add from

25 to 50 cc of tenth-normal alkali, and either boil for one hour with a reflux condenser, cool and titrate with tenth-normal acid, or allow the solution to stand overnight in a stoppered flask with the excess of alkali, heat with a tube condenser for one-half hour at a temperature below the boiling point, cool, and titrate. The number of cubic centimeters of tenth-normal alkali used in the saponification of the esters is calculated as ethyl acetate. One cubic centimeter of tenth-normal alkali equals 0.0088 gram of ethyl acetate.

### 8. Aldehydes.—Provisional.

#### (a) PREPARATION OF REAGENTS.

(1) *Alcohol free from aldehydes*.—Prepare by first redistilling the ordinary 95 per cent alcohol over caustic soda or potash, then add from 2 to 3 grams per liter of m-phenylenediamin hydrochlorid, digest at ordinary temperature for several days (or reflux on the steam bath for several hours) and then distill slowly, rejecting the first 100 cc and the last 200 cc.

(2) *Sulphite-fuchsin solution*.—Dissolve 0.50 gram of pure fuchsin in 500 cc of water, then add 5 grams of  $\text{SO}_2$  dissolved in water, make up to a liter, and allow to stand until colorless. Prepare this solution in small quantities, as it retains its strength for only a very few days.

(3) *Standard acetic aldehyde solution*.—Prepare according to the directions of Vasey<sup>a</sup> as follows: Grind aldehyde ammonia in a mortar with ether and decant the ether. Repeat this operation several times, then dry the purified salt in a current of air and finally in a vacuum over sulphuric acid. Dissolve 1.386 grams of this purified ammonium aldehyde in 50 cc of 95 per cent alcohol, to this add 22.7 cc of normal alcoholic sulphuric acid, then make up to 100 cc and add 0.8 cc to compensate for the volume of the ammonium sulphate precipitate. Allow this to stand over night and filter. This solution contains 1 gram of acetic aldehyde in 100 cc and will retain its strength.

The standard found most convenient for use is 2 cc of this strong aldehyde solution diluted to 100 cc with 50 per cent alcohol by volume. One cubic centimeter of this solution is equal to 0.0002 gram of acetic aldehyde. This solution should be made up fresh every day or so, as it loses its strength.

#### (b) DETERMINATION.

Determine the aldehyde in the distillate prepared for esters. Dilute from 5 to 10 cc of the distillate to 50 cc with aldehyde-free alcohol (50 per cent by volume), add 25 cc of the fuchsin solution, and allow to stand for fifteen minutes at 15° C. The solutions and the reagents should be at 15° C. before they are mixed. Prepare standards of known strength in the same way.

### 9. Furfural.—Provisional.

#### (a) PREPARATION OF REAGENTS.

(1) *Standard furfural solution*.—Prepare by weighing 1 gram of redistilled furfural and dissolving it in 100 cc of 95 per cent alcohol. This strong solution will keep. Standards are made by taking 1 cc of this solution and diluting to 100 cc with 50 per cent by volume alcohol. One cc of this solution contains 0.0001 gram of furfural.

<sup>a</sup> Analysis of Potable Spirits, p. 30.



## (b) DETERMINATION.

Dilute from 10 to 20 cc of the distillate as prepared under ethereal salts to 50 cc with furfural-free alcohol (50 per cent by volume). To this add 2 cc of colorless anilin and 0.5 cc of hydrochloric acid (sp. gr. 1.125) and keep for fifteen minutes in a water bath at about 15° C. Prepare standards of known strength in the same way.

## 10. Fusel Oil.

## (a) ROESE METHOD.—PROVISIONAL.

## (1) PREPARATION OF REAGENTS.

(a) *Fusel-free alcohol*.—Prepare by the fractional distillation of alcohol over caustic alkali, rejecting the first one-fifth and the last three-fifths of the distillate. Dilute to exactly 30 per cent by volume.

(b) *Chloroform*.—Dry and distil.

(c) *Sulphuric acid*.—Specific gravity 1.2857 at 15° C.

## (2) DETERMINATION.

Add a little alkali to 200 cc of the sample under consideration (sufficient to neutralize all the acid and to saponify all the esters, 20 cc normal alkali are usually enough), and distil slowly, until about 175 cc have passed over, allow the distilling flask to cool, add 25 cc of water and distil again until the total distillate measures 200 cc. Dilute the distillate to exactly 30 per cent by volume<sup>a</sup> (sp. gr. 0.96541 at 15.6° C.).

Prepare a water bath, the contents of which are kept at exactly 15° C.; place in it the apparatus (covering the end of the tube with a rubber cap to prevent wetting the inside of the tube) and the flasks which contain the 30 per cent fusel-free alcohol, the chloroform, the sulphuric acid, and the distillate diluted to 30 per cent by volume. When the solutions have all attained the temperature of 15° C. fill the apparatus to the 20-cc mark with the chloroform, drawing it through the lower tube by means of suction, add 100 cc of the 30 per cent fusel-free alcohol and 1 cc of the sulphuric acid, invert the apparatus and shake vigorously for two or three minutes, interrupting once or twice to open the stopcock for the purpose of equalizing pressure. Allow the apparatus to

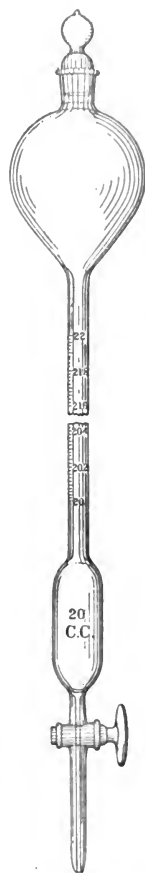


FIG. 3.—Bromwell's fusel oil apparatus.

<sup>a</sup> The following is an accurate method for diluting any given alcohol solution to a weaker solution of definite percentage: Designate the volume percentage of the stronger alcohol by  $V$  and that of the weaker alcohol by  $v$ . Mix  $v$  volumes of the stronger alcohol with water to make  $V$  volumes of the product. Allow the mixture to stand till full contraction has taken place and till it has reached the temperature of the original alcohol and water and make up any deficiency in the  $V$  volumes with water.

*Example*.—It is desired to dilute a distillate containing 50 per cent of alcohol by volume until it contains 30 per cent. To 30 volumes of the 50 per cent alcohol add enough water to make 50 volumes, or place 150 cc of the distillate in a 250-cc flask, fill to the mark with water, mix, cool, and fill to the mark again.

Owing to the extreme difficulty of preparing distillates of exactly 30 per cent, slight variations may be corrected by increasing or decreasing the chloroform reading, as suggested by Sell, 0.003 cc for each 0.01 per cent variation in strength of alcohol from 30 per cent. Such variation, however, should not exceed 0.02 per cent.

stand for one hour in water that is kept at 15° C.<sup>a</sup> turning occasionally to hasten the settling of the chloroform, and note the volume of the chloroform. After thoroughly cleansing and drying the apparatus repeat this operation, using the diluted distillate from the sample under examination instead of the fusel-free alcohol. The increase in the chloroform volume with the samples under examination over that with fusel-free alcohol is due to fusel oil, and this difference (expressed in cubic centimeters) multiplied by the factor 0.663 gives the volume of fusel oil in 100 cc, which is equal to the percentage of fusel oil by volume in the 30 per cent distillate. This must be calculated to the percentage of fusel oil by volume in the original liquor.

*Example.*—A sample of liquor contains 50 per cent of alcohol by volume. The increase in the chloroform volume with the 30 per cent fusel-free alcohol is 1.42 cc. The increase in the chloroform volume with the distillate from the liquor under examination diluted to 30 per cent is 1.62 cc; difference, 0.20 cc. The volume of fusel oil in 100 cc of the 30 per cent distillate is then  $0.20 \times 0.663 = 0.1326$ , and the percentage of fusel oil by volume in the original liquor is  $\frac{50 \times 0.1326}{30} = 0.221$ .

#### (b) ALLEN-MARQUARDT METHOD.—PROVISIONAL.

Add to 100 cc of whisky 20 cc of half-normal sodium hydroxid, and saponify the mixture by boiling for one hour under a reflux condenser.<sup>b</sup> Connect the flasks with a distilling apparatus, distil 90 cc, add 25 cc of water, and continue the distillation until an additional 25 cc is collected.

Approximately saturate the distillate with finely ground sodium chlorid and add a saturated solution of sodium chlorid until the specific gravity is 1.10.

Extract this salt solution four times with carbon tetrachlorid,<sup>c</sup> using 40, 30, 20, and 10 cc, respectively, and wash the carbon tetrachlorid three times with 50 cc portions of a saturated solution of sodium chlorid and once with saturated solution of sodium sulphate. Then transfer the carbon tetrachlorid to a flask containing 5 cc of concentrated sulphuric acid, 45 cc of water, and 5 grams of potassium bichromate, and boil for eight hours under a reflux condenser.

Add 30 cc of water, and distil until only about 20 cc remain; add 80 cc of water, and distil until but 5 cc are left. Neutralize the distillate to methyl orange, and titrate with sodium hydroxid, using phenolphthalein as indicator. One cubic centimeter of tenth-normal sodium hydroxid is equivalent to 0.0088 gram of amyl alcohol.

Rubber stoppers can be used in the saponification and first distillation, but corks covered with tinfoil must be used in the oxidation and second distillation. Corks and tinfoil must be renewed frequently.

### 11. Sugar.—Provisional.

Proceed as directed under "XIII. Wine," page 87.

<sup>a</sup> The temperature must be held as nearly 15° C. as possible. If any variations occur, the chloroform must be increased or decreased 0.046 cc for every degree above or below that temperature (Gebek und Stutzer, Zts. angew. Chem., 1893, 132).

<sup>b</sup> Or 100 cc of the liquor may be mixed with 20 cc of half-normal sodium hydroxid, allowed to stand overnight at room temperature, and distilled directly.

<sup>c</sup> Purify 5 liters of carbon tetrachlorid by boiling for several hours under a reflux condenser with 200 cc of sulphuric acid and 25 grams of potassium bichromate in 200 cc of water; separate from the oxidizing mixture by distillation, and redistil over barium carbonate.

## 12. Detection of Methyl Alcohol.

(a) TRILLAT METHOD.<sup>a</sup>—PROVISIONAL.

To 50 cc add 50 cc of water and 8 grams of lime, and fractionally distil by the aid of Glinsky bulb tubes. Dilute the first 15 cc of the distillate to 150 cc, mix with 15 grams of potassium bichromate and 70 cc of sulphuric acid (1 : 5), and allow to stand for one hour with occasional shaking.

Distil, reject the first 25 cc, and collect 100 cc. Mix 50 cc of the distillate with 1 cc of rectified dimethyl-anilin, transfer to a stout, tightly stoppered flask, and keep on bath at 70° to 80° C. for three hours with occasional shaking. Make distinctly alkaline with sodium hydroxid, and distil the excess of dimethyl-anilin, stopping the distillation when 25 cc have passed over.

Acidify the residue in the flask with acetic acid, shake, and test a few cubic centimeters by adding 4 or 5 drops of water with lead dioxid in suspension (1 gram in 100 cc). If methyl alcohol be present, a blue coloration occurs which is increased by boiling.

NOTE.—Ethyl alcohol thus treated yields a blue coloration changing immediately to green, afterwards to yellow, and becoming colorless when boiled.

(b) RICHE AND BARDY METHOD.<sup>b</sup>—PROVISIONAL.

The following method for the detection of methyl alcohol in commercial spirit of wine depends on the formation of methyl-anilin violet:

Place 10 cc of the sample, previously rectified over potassium carbonate if necessary, in a small flask with 15 grams of iodine and 2 grams of red phosphorus. Keep in ice water for from ten to fifteen minutes until action has ceased. Distil on a water bath the methyl and ethyl iodids formed into about 30 cc of water. Wash with dilute alkali to eliminate free iodine. Separate the heavy oily liquid which settles and transfer to a flask containing 5 cc of aniline. The flask should be placed in cold water, in case the action should be violent, or, if necessary, the reaction may be stimulated by gently warming the flask. After one hour boil the product with water and add about 20 cc of a 15 per cent solution of soda; when the bases rise to the top as an oily layer fill the flask up to the neck with water and draw them off with a pipette. Oxidize 1 cc of the oily liquid by adding 10 grams of a mixture of 100 parts of clean sand, 2 of common salt, and 3 of cupric nitrate; mix thoroughly, introduce into a glass tube, and heat to 90° C. for eight or ten hours. Exhaust the product with warm alcohol, filter, and make up with alcohol to 100 cc. If the sample of spirits be pure the liquid is of a red tint, but in the presence of 1 per cent of methyl alcohol it has a distinct violet shade; with 2.5 per cent the shade is very distinct, and still more so with 5 per cent. To detect more minute quantities of methyl alcohol, dilute 5 cc of the colored liquid to 100 cc with water, and dilute 5 cc of this again to 400 cc. Heat the liquid thus obtained in porcelain and immerse a fragment of white merino (free from sulphur) in it for half an hour. If the alcohol be pure the wool will remain white, but if methylated the fiber will become violet, the depth of tint giving a fair approximate indication of the proportion of methyl alcohol present.

<sup>a</sup> A. Trillat, *Analyst*, 1899, 24: 13, 211–212.

<sup>b</sup> Allen's *Commercial Organic Analysis*, 3d ed., 1: 80.



**13. Detection of Coloring Matter.—Provisional.**

In general proceed as directed under "XXVIII. Coloring Matter," page 190.

**(a) CRAMPTON AND SIMONS TEST FOR CARAMEL.<sup>a</sup>**

This method depends on the insolubility of caramel in ether. Evaporate 50 cc of the sample nearly to dryness on the water bath, wash into a 50 cc flask, add 25 cc of absolute alcohol, cool to a definite temperature, and dilute to mark with water. Transfer 25 cc to an apparatus of the general description of Bromwell's fusel-oil apparatus (p. 97), but graduated so that the lower bulb holds 25 cc to a definite mark on the stem, which may be of larger bore than in Bromwell's apparatus. Add 50 cc of ether and shake at intervals for half an hour, let settle, and siphon water through the lower tube until the aqueous layer reaches the 25 cc mark. Mix the whole, remove the aqueous layer, and compare with the 25 cc of the solution which were not treated with ether. Express the amount of color removed on percentage basis.

**(b) AMTHOR TEST MODIFIED BY LASCHÉ.<sup>b</sup>—PROVISIONAL.**

Add 10 cc of paraldehyde to 5 cc of the sample contained in a test tube and shake. Add absolute alcohol, a few drops at a time, shaking after each addition until the mixture becomes clear. Allow to stand. Turbidity after ten minutes is an indication of caramel.

<sup>a</sup> J. Amer. Chem. Soc., 1890, 22: 810.

<sup>b</sup> The Brewer Distiller, May, 1903.

## **XVI. METHODS FOR THE ANALYSIS OF VINEGAR.—PROVISIONAL.**

### **1. Preparation of Sample.**

For microscopical examinations employ the original sample but for chemical analysis filter if turbid. Keep the samples in glass-stoppered bottles and analyze promptly to avoid fermentation.

### **2. Calculation of Results.**

Express all results as per cent by weight. For most purposes cubic centimeters may be regarded as grams; if greater accuracy is desired weigh or calculate weight from specific gravity.

### **3. Specific Gravity.**

Determine as directed under "XIII. Wine," page 83.

### **4. Alcohol.**

Carefully neutralize 100 cc and distil 40 cc; redistil until 20 cc have passed over; cool to 15.5° C. and make up to 20 cc with distilled water. Determine the specific gravity by means of a pycnometer and calculate the percentage by weight from Table II, page 203.

### **5. Solids.**

Evaporate 10 cc to a sirupy consistency on a water bath in a tared platinum dish of 50 mm diameter, dry for two and one-half hours in the drying oven at the temperature of boiling water, cool, and weigh.

### **6. Ash.**

Proceed as directed under "VI. General Methods," page 38, employing the residue from the determination of the solids, according to the preceding section.

### **7. Solubility and Alkalinity of the Ash.**

Ash 25 cc of the vinegar, extract the ash repeatedly with hot water on an ashless filter, dry and ignite the filter with the undissolved residue, cool, weigh, and calculate as insoluble ash. Titrate the aqueous extract with tenth-normal acid, using methyl orange as indicator.

### **8. Phosphoric Acid of the Ash.**

Determine in the water-soluble and water-insoluble portions by the official method, page 2, under "I. Fertilizers." Express the results as milligrams of phosphoric acid in 100 grams of vinegar.

### 9. Total Acids.

Titrate a suitable amount of the sample, which has been diluted until it appears very slightly colored, with standard alkali, using phenolphthalein as indicator. One cubic centimeter of tenth-normal alkali is equivalent to 0.0060 gram of acetic acid.

### 10. Volatile Acids.

Heat 15 cc to boiling in a flask, adding a little tannin if foaming occurs; then lower the flame and pass a current of steam through the vinegar to a condenser. Continue the operation until 15 cc of distillate shows no acidity with sensitive litmus paper. Titrate the combined distillate as directed for total acids.

### 11. Fixed Acids.

Deduct volatile acids from total acids and multiply the remainder by 0.817 for sulphuric acid, or 1.117 for malic acid. Or, in case tannin has not been added, dilute the nonvolatile residue from the distillation with recently boiled water, and titrate with standard alkali as directed for total acid.

One cubic centimeter of tenth-normal alkali equals 0.0049 gram of sulphuric acid, or 0.0067 gram of malic acid.

### 12. Oxalic Acid.

Oxalic acid may be detected and determined by precipitation with calcium sulphate.

### 13. Detection of Bitartrate of Potassium—Allen's Method.<sup>a</sup>

Treat the residue, left from evaporation of the vinegar, with alcohol; a granular residue of tartar remains undissolved; to prove its character, pour off the alcohol and dissolve the residue in a small quantity of hot water. On cooling the aqueous solution and rubbing the sides of the vessel with a glass rod the acid tartrate of potassium deposits in streaks. An addition of an equal bulk of alcohol makes the reaction more delicate.

### 14. Free Tartaric Acid.

Treat the alcoholic solution of the extract with an alcoholic solution of potassium acetate. If tartaric acid be present streaks will form on the beaker on stirring with a glass rod, or sometimes a distinct precipitate will separate. Approximately quantitative results can be obtained by titration of the precipitate with standard alkali.

### 15. Detection of Free Mineral Acids.

#### (a) LOGWOOD METHOD.<sup>b</sup>

Prepare an extract of logwood by pouring 100 cc of boiling water upon 2 grams of fresh logwood chips, allowing the decoction to stand for a few hours, and filtering. Place drops of the liquid on a porcelain surface and dry in a water bath. Add to one of the spots a drop of the vinegar to be tested (after concentration if desirable), and evaporate to dryness. A yellow tint remains if free mineral acids are absent, a red tint if present.

<sup>a</sup> Allen's Commercial Organic Analysis, 2d ed., 1: 389.

<sup>b</sup> Ashby, Allen's Commercial Organic Analysis, 2d ed., 1: 393.

## (b) METHYL-VIOLET METHOD.

Add to 5 cc of vinegar 5 or 10 cc of water; after mixing well, add 4 or 5 drops of an aqueous solution of methyl-violet (one part of methyl-violet 2B in 10,000 parts of water). The occurrence of a blue or green color indicates the presence of a free mineral acid.

**16. Determination of Free Mineral Acids.**

## (a) HILGER'S METHOD.

Exactly neutralize 20 cc with half-normal alkali, using sensitive neutral litmus paper as indicator. Evaporate to one-tenth of its volume in a porcelain dish, add a few drops of methyl-violet (see sec. 15, preceding), dilute with 3 or 4 cc of water if necessary to secure a clear solution, bring to boiling, and titrate with half-normal sulphuric acid until a green or blue color begins to appear. The difference, in cubic centimeters, between the half-normal alkali and the acid added, multiplied by the factor 0.1225, expresses the percentage of mineral acid present in terms of sulphuric acid ( $H_2SO_4$ ).

## (b) HEHNER'S METHOD.

To a weighed quantity of the sample add excess of tenth-normal alkali, evaporate to dryness, incinerate, and titrate the ash with tenth-normal acid, using methyl-orange as indicator. The difference between the number of cubic centimeters of alkali added in the first place and the number of cubic centimeters of acid needed to titrate the ash represents the free mineral acid present.

**17. Reducing Sugar After Inversion.**

Determine after inversion as directed under "XIII. Wine," page 87, except that it is not necessary to evaporate to remove alcohol.

**18. Polarization.**

Polarize in a 400 mm tube, preparing solution as directed for reducing sugars, under "VI. General Methods," page 40.

**19. Detection of Dextrin.**

Dextrin is often found in glucose vinegar, and is precipitated from the concentrated vinegar upon addition of three or four volumes of strong alcohol; the precipitate may be identified by its physical properties and the color reaction with iodine solution.

**20. Detection of Coloring Matters.**

The principal coloring matter used for tinting imitation vinegars in America is caramel. To detect this use Amthor's method as given under "XV. Distilled Liquors," page 101.

A further test for caramel may be made by boiling the aqueous solution of a portion of the precipitate obtained by Amthor's method, with Fehling's solution; caramel has a reducing action.

In the case of wine vinegars, a test for foreign red colors may be made according to the methods given under "XXVIII. Coloring Matter," page 190.



**21. Lead Acetate Precipitate.**

Add a few drops of neutral lead acetate to 10 cc of a vinegar. Failure to obtain a precipitate proves that it is not a cider vinegar.

**22. Detection of Foreign Pungent Materials.**

Exactly neutralize a portion of the vinegar (the residue from determination of total acidity may be used), evaporate part of the water, and test the concentrated solution by taste for pungent substances; agitate the liquid with ether in a separatory funnel, remove and evaporate the ethereal layer, and taste the residue. The identification of the specific substance is rarely possible.

**23. Heavy Metals.**

Evaporate from 200 to 500 cc to dryness. In case of cider, malt, and other vinegars rich in solids, add a little sodium hydroxid and potassium nitrate and incinerate. Dissolve the ash thus obtained, or the solids themselves, and proceed as directed under "IX. Canned Vegetables," page 61.

**24. Detection of Preservatives.**

Proceed as directed under "XXVII. Food Preservatives," page 179.

## **XVII. METHODS FOR THE ANALYSIS OF MEAT AND MEAT PRODUCTS.<sup>a</sup>**

### **MEAT.**

#### **1. Identification of Species.—Provisional.**

The percentage of glycogen, added to the percentage of reducing sugar, is often of value in detecting horse meat. Certain results obtained in the examination of the fat also afford valuable data. Among the factors which are of value for this purpose may be mentioned the iodine number, melting point, congealing point, index of refraction, and to a less extent the specific gravity, acetyl number, and Maumené value. The meat from embryonic animals and from animals killed before they are suitable for food may often be detected by its moist, clammy nature and high water content. The subject of the determination of the species of animal from which meat is taken is well treated in works on meat inspection and is not discussed here.

#### **2. Injurious and Poisonous Products.<sup>b</sup>—Provisional.**

The ordinary foods of man are liable to become unwholesome or poisonous from any of the following causes: (1) *Trichinæ* in pork, (2) metals, and (3) bacterial products.

##### **(a) TRICHINÆ.**

Examine immediately for *trichinæ* <sup>c</sup> pork, or sausage containing pork, which has caused sickness.

##### **(b) POISONOUS METALS.**

Examine for arsenic, antimony, tin, lead, copper, and zinc. For the determination of these metals proceed as directed under "IX. Canned Vegetables," page 61.

##### **(c) BACTERIAL PRODUCTS.**

As bacteria are the most common cause of poisonous meat, the examination should primarily be made from that point of view. Given a poisonous meat, the first procedure is to examine for *trichinæ*. If they are not found, the bacteriological examination should next be undertaken and the chemical examination reserved until the last.

The bacteriological examination should first consist in feeding a number of different species of animals—the larger the number the better—for a day or two exclusively upon the food. White mice, house mice, white rats, young dogs,

<sup>a</sup> See Appendix, p. 252, for method for the determination of acidity.

<sup>b</sup> The material under this heading was originally prepared by Dr. E. C. Novy, of Ann Arbor, Mich.

<sup>c</sup> Fishchöder, *Leitfaden der praktischen Fleischschau*; Ostertag, *Handbuch der Fleischschau*; Walley, *A Practical Guide to Meat Inspection*, pp. 233-245.

cats, rabbits, or guinea pigs may be used. If the animals sicken and die, they are to be subsequently examined for the presence of pathogenic bacteria. It may happen that none of the animals thus fed will be injured by the food, which fact would not exclude, however, the presence of a germ requiring a specially susceptible animal for a subject.

Another set of animals should be injected with a cold extract of the meat made with sterile water. If the animals die, they are to be examined for pathogenic bacteria. A third set of animals should receive similar injections, though of larger portions, of this aqueous extract which has been previously filtered through sterile porcelain. If the animals die from such injections, the same as with unfiltered solutions, it is evident that a soluble bacterial chemical poison is present.

The identification of the toxin produced by the germ is wholly out of the question. The most that can be done satisfactorily is to obtain, as above, a germ-free solution of the poison. It is wholly unnecessary to devote any space in this connection to the detection of the basic bacterial products, the ptomaines, since these bodies are mere cleavage products produced by some and not by other bacteria. Moreover, they are usually but very feebly poisonous, and therefore are not considered to be as important as formerly.<sup>a</sup>

A bacteriological examination proper should be made of the original poisonous meat and of all the animals that died either from eating the meat or from the injections of the aqueous extracts. The organism present in the animal, if any, must be isolable directly from the meat. If it happens, as it sometimes has, that the dead animals contain no germs, it is proof that they were killed by a toxin elaborated by a germ in the meat previous to the injection. Cultures from the meat will then reveal the germ, and the effects of its pure cultures should correspond to those observed with the poisonous meat.

To prepare the cultures from the original food, the latter should be cut out with a sterile knife and material should be taken from the inside, thus avoiding all chances of contamination. Several sets of beef-tea tubes and agar plates should be made. One set should be set aside in a Novy anaerobic jar at room temperature; a second similar set should be placed at a temperature of 37° C. A third set should be grown in the presence of air at room temperature, and a like set at a temperature of 37° C.

The full details of bacteriological methods must obviously be omitted in this connection. Such work requires a special laboratory and special drill. Those who may be further interested are referred to the works of Abbott, Novy, and Sternberg.

### 3. Preparation of Sample for Chemical Examination.—Provisional.

In the case of fresh meat, separate the sample as completely as possible from the bones and pass it rapidly and repeatedly through a sausage mill until thorough mixture and complete maceration are obtained. The sample must be kept on ice to prevent decomposition, and all of the determinations should be begun as soon as practicable after the sample is prepared. In the case of canned meats, pass the entire contents of a can through a sausage mill as directed above. Remove sausage from the casings and mix by repeated grinding in a sausage mill. Dry that portion of the sample which is not needed for analysis, extract with gasoline which boils below 60° C., allow the gasoline to evaporate spontaneously, and expel the last traces by heating for a short time on the steam bath. Neither the meat nor the separated fat should be heated longer than

<sup>a</sup> For detailed methods see Vaughan and Novy, "Cellular Toxins," 4th edition, 1902.

necessary, owing to the tendency of the latter to decompose. Reserve the fat for examination according to the methods given under "XIX. Edible Oils and Fats," page 129. Fat must be kept in a cool place and the examination finished before it becomes rancid.

#### 4. Moisture.—Provisional.

Proceed as directed under "VI. General Methods," page 38.

#### 5. Ash.—Provisional.

Proceed as directed under "VI. General Methods," page 38.

#### 6. Ether Extract or Crude Fat.—Provisional.

Proceed as directed under "VI. General Methods," page 39.

#### 7. Nitrogenous Substances.—Provisional.

##### (a) TOTAL NITROGEN.

Employ either the Kjeldahl or the Gunning method (as given under "I. Fertilizers," p. 5) using about 2 grams of the fresh sample. The digestion with sulphuric acid should be continued at least four hours.

##### (b) INSOLUBLE PROTEIDS.

Thoroughly exhaust 2 grams of the sample with cold water after extraction with ether, filter, and determine nitrogen in the insoluble residue as directed under "(a) Total nitrogen." Multiply the percentage of nitrogen so obtained by 6.25 for the percentage of meat fiber or insoluble proteids. (In case the connective tissue is determined, a corresponding correction must be made in the percentage of insoluble proteids.)

##### (c) CONNECTIVE TISSUE.

Exhaust 10 grams with cold water as directed above, then boil the exhausted residue repeatedly with about 100 cc of water until the total extract amounts to approximately 1 liter. Filter, concentrate by evaporation, and determine the nitrogen content. Multiply the nitrogen so obtained by 5.55 to obtain the percentage of nitrogenous substances of connective tissue.

##### (d) COAGULABLE PROTEIDS.

(For uncooked meat only.)

Almost neutralize the filtrate from the insoluble proteids, leaving it still faintly acid, boil until the coagulable proteids separate, filter, wash, transfer the filter paper and contents to a Kjeldahl flask, and determine nitrogen as directed above under "(a) Total nitrogen." Multiply the percentage of nitrogen obtained by 6.25 to obtain the percentage of coagulable proteids.

##### (e) PROTEOSES, PEPTONES, AND GELATIN.

Heat the filtrate from albumin and globulins, add a slight excess of tannic acid and a few drops of a saturated solution of alum, allow to cool, filter, and wash with cold water. Heat the filtrate from the tannic-acid precipitate almost

to boiling, add an excess of phosphotungstic acid,<sup>a</sup> separate the precipitated proteids by filtration and wash with hot water, being careful that the temperature of the solution and wash water shall not be less than 90° C. at any time.

Transfer the filter papers containing the tannic acid and phosphotungstic acid precipitates to a Kjeldahl flask and determine nitrogen. The nitrogen so obtained multiplied by 6.25 gives the percentage of proteoses, peptones, and gelatin.

#### (f) MEAT BASES.

Deduct from the total nitrogen the sum of the nitrogen obtained in the determination of insoluble proteids, coagulable proteids, proteoses, peptones, and gelatin for the nitrogen of the meat bases. Multiply the result by 3.12 to obtain the percentage of meat bases.

### 8. Starch.

(In chopped meat, sausage, deviled meat, etc.)

#### (a) QUALITATIVE DETERMINATION.—PROVISIONAL.

Treat 5 or 6 grams of the sample with boiling water for two or three minutes; cool the mixture, and test the supernatant liquid with iodine solution. In using this test it must be remembered that a small amount of starch may be present as the result of the use of spices. If a marked reaction is given, however, it may be concluded that starch or flour has been added, and a quantitative determination may be made. The above qualitative method may be replaced by a microscopic examination, by which not only the presence of added starch, but also the variety employed may be determined.

#### (b) QUANTITATIVE DETERMINATION.

##### (1) AMBÜHL'S METHOD.<sup>b</sup>—PROVISIONAL.

This method is short and convenient, but the results obtained by it are only roughly approximate.

Thoroughly macerate 2 grams<sup>c</sup> of the meat under examination with 50 times its weight of water. Boil for thirty minutes and dilute to 100 cc for every gram of meat employed. Cool an aliquot of the liquid, treat with iodine, and compare the depth of color with solutions containing a known amount of the same kind of starch (the variety of starch in the sample is determined microscopically), and boiled for the same length of time.

##### (2) MAYRHOFFER'S METHOD<sup>d</sup> MODIFIED.—PROVISIONAL.

Treat from 10 to 20 grams of the sample under examination (depending upon the amount of starch indicated by the iodine reaction) in a porcelain dish or

<sup>a</sup> Mallet employs two solutions, one containing 50 and the other 100 grams of crystalline phosphotungstic acid dissolved in 1 liter of 2.5 per cent hydrochloric acid. He also recommends the addition of sand or pulverized glass to prevent the formation of coagulated proteids in a dense clot. Owing to the liability of "bumping" in the presence of such substances, however, during the determination of nitrogen it would seem that such addition should be avoided if possible.

<sup>b</sup> Pharm. Centralh., 1881, 22: 438; Abstract Zts. anal. Chem., 1882, 21: 436.

<sup>c</sup> Ambühl directs that from 2 to 10 grams be employed, according to the size of the meat particles. If the sample be macerated, however, as directed under the preparation of sample, it is unnecessary to employ a large amount.

<sup>d</sup> Forschungs-Ber. Lebensm., 1896, 3: 141; Ibid., 1897, 4: 47.

casserole with 50 cc of an 8 per cent solution of potassium hydroxid and heat the mixture on the water bath until the meat is entirely dissolved. The operation may be hastened by macerating the larger pieces with a glass rod. Add an equal volume of 95 per cent alcohol, mix thoroughly, filter through an asbestos filter, and wash twice with a hot 4 per cent solution of potassium hydroxid in 50 per cent alcohol. Then wash with 50 per cent alcohol until a small portion of the filtrate does not become turbid upon the addition of acid. Return the precipitate and filter to the original vessel and dissolve, with the aid of heat, in 60 cc of a normal solution of potassium hydroxid. In the case of sausage with a high starch content a somewhat larger volume of alkali may be required. Acidify the filtrate strongly with acetic acid, dilute to a definite volume, thoroughly mix by shaking, filter through a folded filter, and precipitate the starch from an aliquot of the filtrate by means of an equal volume of 95 per cent alcohol. Transfer the precipitate to a filter, thoroughly wash with 50 per cent alcohol, with absolute alcohol, and finally with ether, dry to a constant weight at the temperature of boiling water, and weigh.

### 9. Glycogen.

The determination of glycogen<sup>a</sup> has been suggested as a means of detecting the presence of horse meat. Subsequent results indicate that this determination is of limited value because of the fact that glycogen begins to disappear soon after the death of the animal, and may entirely disappear after a short lapse of time. No definite conclusions can therefore be derived from the results of this determination, but it is of value as a confirmatory test.

#### (a) QUALITATIVE METHOD.<sup>b</sup>—PROVISIONAL.

Boil 50 grams of the macerated sample with 50 cc of water for from fifteen to thirty minutes. Filter the broth through moistened filter paper or fine linen. To a portion of the filtrate in a test tube add a few drops of a mixture of 2 parts iodine, 4 parts potassium iodide, and 100 parts water. In the presence of a large percentage of horse meat the glycogen contained produces a dark-brown color, which is destroyed by heating and reappears on cooling. When starch is present it may be precipitated by two volumes of glacial acetic acid, separated by filtration, and the test for glycogen repeated in the filtrate.

#### (b) QUANTITATIVE METHOD.<sup>c</sup>—PROVISIONAL.

Digest 50 grams of finely macerated meat on the water bath with 200 cc of 2 per cent potassium hydroxid until solution is practically complete. Cool, dilute with water to exactly 200 cc, shake, and filter. Treat 100 cc of the filtrate with 10 grams of potassium iodide and 1 gram of potassium hydroxid and stir until solution is complete. Add 50 cc of 96 per cent alcohol and allow to stand until the following day. Separate the precipitated glycogen by filtration and wash with a solution containing 1 cc of 73 per cent potassium hydroxid, 10 grams of potassium iodide, 100 cc of water, and 50 cc of 96 per cent alcohol. Wash the glycogen with a mixture of two volumes of 96 per cent alcohol and one volume of water (the mixture containing about 7 mg of sodium chloride per liter), dissolve in water, and remove the remaining traces of proteids by the addition of double iodide of mercury and potassium. It is

<sup>a</sup> Niebel, *Abh. Zts. angew. Chem.*, 1895, 620.

<sup>b</sup> Courlay and Coremons, *Abh. Zts. Nahr. Waar.*, 1896, 10: 173.

<sup>c</sup> Pflüger und Nerking, *Arch. gesam. Physiol.*, 1899, 76: 531.

often found that the proteids are so completely removed that no precipitate is formed with the double iodid, and filtration is not necessary.

Add about 2 mg of sodium chlorid per 100 cc of water, precipitate the glycogen again by means of two volumes of 96 per cent alcohol, filter, wash with 96 per cent alcohol, containing about 7 mg of sodium chlorid per liter, then with absolute alcohol, finally with ether, dry to constant weight, and weigh.

As a control, invert the precipitated glycogen by boiling for three hours with hydrochloric acid, diluted with 10 parts of water, and determine the reducing sugar by Allihn's method, multiplying the result by 0.9 to obtain the percentage of glycogen.

#### 10. Reducing Sugar.—Provisional.

Boil 100 grams of the finely divided meat for fifteen or twenty minutes in a 500 cc graduated flask, with a convenient volume of water. Add a few cubic centimeters of normal lead acetate, cool to room temperature, make up to the mark with water, and filter through a folded filter. Remove the lead and determine reducing sugar as dextrose, as described under "VI. General Methods," page 49.

#### 11. Potassium Nitrate.

##### (a) SCHLÖSING-WAGNER METHOD.<sup>a</sup>—PROVISIONAL.

Use a flask of about 250 cc capacity with a rubber stopper having two holes. Through one of them pass the stem of a funnel carrying a glass stopcock; the other carries a delivery tube leading to the receiving vessel. Bend the end of the delivery tube so that it will pass easily under the mouth of the measuring burette and cover with a piece of rubber tubing.

Place 50 cc of saturated ferrous chlorid solution and the same quantity of 10 per cent hydrochloric acid in a flask. Prepare the ferrous chlorid solution by dissolving nails or other small pieces of iron in hot hydrochloric acid and keep in glass-stoppered flasks of about 50 cc capacity, entirely filled. The contents of one flask is enough for about twelve determinations, and by using the entire contents of a flask as soon as possible after opening all danger of oxidation is avoided.

Boil the contents of the flask until the air is driven off; then place the delivery tube under the measuring tube, which is filled with 40 per cent potassium hydroxid, add a few drops of water, and cover the tube with pieces of filter paper. By a careful and quick inversion the measuring tube can be brought into the vessel receiving it without any danger of air entering.

Extract 100 grams of finely macerated meat by boiling repeatedly with successive small portions of water, concentrate the aqueous solution to a small volume, transfer to the funnel, and, with continued boiling, allow it to pass drop by drop into the flask. When the solution has almost all run out, wash the funnel with three 10 cc portions of 10 per cent hydrochloric acid and allow these portions to pass drop by drop into the flask. The temperature of the surrounding water will soon be imparted to the contents of the tube, and the volume of nitric oxid is read with the tube in such a position that the level of the water within and without the tube coincides.

The amount of nitric oxid present and the corresponding percentage of nitrate may be calculated in the usual way for the given temperature and barometric pressure, or, to avoid computation, the amount of nitrate may be determined

<sup>a</sup> Bieler and Schneidewind, Die agrikultur-chemische Versuchsstation Halle a/S, 1892, p. 50; Wiley, Principles and Practice of Agricultural Analysis, 1897, 2: 228.

by comparison of the volume of nitric oxid with that evolved by a definite volume (5 to 10 cc) of normal sodium nitrate solution.

(b) PHENOL-SULPHONIC ACID METHOD.<sup>a</sup>—PROVISIONAL.

Weigh 1 gram of the sample into a 100 cc flask, add from 20 to 30 cc of water, and heat on the water bath for fifteen minutes, shaking occasionally. Add 3 cc of a saturated solution of silver sulphate for each per cent of sodium chlorid present, then add 10 cc of lead subacetate and 5 cc of alumina cream, shaking after each addition. Make up to mark with water and filter through a folded filter, returning the filtrate to the filter until it runs clear. Evaporate to dryness 25 cc of the filtrate, add 1 cc of phenol-sulphonic acid,<sup>b</sup> mix thoroughly with a glass rod, add 1 cc of water and 3 or 4 drops of concentrated sulphuric acid, and heat on a steam bath for two or three minutes, being careful not to raise the temperature sufficiently to char the material. Add about 25 cc of water and an excess of ammonium hydroxid. Transfer to a 100 cc flask, add 1 or 2 cc of alumina cream if not perfectly clear, dilute to the mark with water, and filter if necessary.

Prepare a number of 50-cc Nessler tubes, preferably the long, narrow tubes, placing in the first 1 cc of the standard nitrate solution, containing 0.01 mg of nitrogen as potassium nitrate in each cubic centimeter, in the second 2 cc, and so on to 10 cc, then 12 cc, 15 cc, 18 cc, and 20 cc. Compare with the standards the solution prepared as directed above. If dilution is necessary to bring within this range, calculate to original concentration, and note in the following table the percentage of potassium nitrate in the original sample.

*Per cent potassium nitrate.*

Standard solution.	Potassium nitrate.	Standard solution.	Potassium nitrate.	Standard solution.	Potassium nitrate.
cc.	Per cent.	cc.	Per cent.	cc.	Per cent.
0.7	0.01	14.7	0.21	28.7	0.41
1.4	.02	15.4	.22	29.4	.42
2.1	.03	16.1	.23	30.1	.43
2.8	.04	16.8	.24	30.8	.44
3.5	.05	17.5	.25	31.5	.45
4.2	.06	18.2	.26	32.2	.46
4.9	.07	18.9	.27	32.9	.47
5.6	.08	19.6	.28	33.6	.48
6.3	.09	20.3	.29	34.3	.49
7.0	.10	21.0	.30	35.0	.50
7.7	.11	21.7	.31	35.7	.51
8.4	.12	22.4	.32	36.4	.52
9.1	.13	23.1	.33	37.1	.53
9.8	.14	23.8	.34	37.8	.54
10.5	.15	24.5	.35	38.5	.55
11.2	.16	25.2	.36	39.2	.56
11.9	.17	25.9	.37	39.9	.57
12.6	.18	26.6	.38	40.6	.58
13.3	.19	27.3	.39	41.3	.59
14.0	.20	28.0	.40	42.0	.60

12. Detection of Preservatives.—Provisional.

The chemical preservatives commonly used with meat products are borax and boric acid and sulphites. Salicylic and benzoic acids are occasionally used. The methods for the detection of these preservatives are given under "XXVII. Food

<sup>a</sup> This method is a modification of the one ordinarily employed for determining potassium nitrate in water. It was adapted to the examination of meat by Arthur Given.

<sup>b</sup> Prepared by mixing 37 cc of concentrated sulphuric acid, 3 cc of distilled water, and 6 grams of phenol.



Preservatives," page 179. In general, preservatives may be separated from meat by digesting a few minutes in warm water made slightly acid or slightly alkaline, according as the nature of the preservative is basic or acid.

#### (a) SULPHUROUS ACID.

The distillation method for the detection of sulphurous acid (see page 187, under "XXVII. Food Preservatives") may be employed for the examination of meat, using 20 cc instead of 5 cc of the 20 per cent solution of phosphoric acid. Mere traces should be ignored. According to Ostertag<sup>a</sup> the microscopic examination of meat that has been preserved with sodium or calcium sulphite often discloses the presence of crystals of sodium or calcium sulphate, due to partial oxidation of the sulphite.

In the absence of chlorids and nitrates, Kämmerer<sup>a</sup> employs potassium iodate paper in the following manner: Place the sample of meat on potassium iodate paper and moisten it with dilute sulphuric acid (1:8) free from oxids of nitrogen. In the presence of even minute traces of sulphites a deep blue color is immediately formed, while in the absence of sulphites only a faint blue color is produced, and that after a considerable time. This method is of limited application, since it can not be used with meats containing salt or saltpeter.

### 13. Detection of Coloring Matter.—Provisional.

Sausages and other preparations in which chopped meat is employed rapidly became discolored on exposure to the air. This change does not take place to a marked extent with meat that has been cured in a pickle containing saltpeter. With fresh chopped meat, and sometimes with corned meat, especially that cured without saltpeter, coloring matter is sometimes added to prevent the change of color which would naturally take place. The coloring matter may often be extracted by heating for fifteen or twenty minutes with 50 per cent alcohol, with 50 per cent glycerin slightly acidified, with a mixture of alcohol and glycerin,<sup>b</sup> with ammonium hydroxid, or with a 5 per cent solution of sodium salicylate<sup>c</sup> in water. Approximately equal weights of meat and solvent may be used.

In case the filtered extract by any of these methods is colored red or deep yellow, evaporate it nearly to dryness, slightly acidify with hydrochloric acid, and boil a few minutes after the addition of a thread of fat-free wool. If the wool is dyed, examine it as directed under "XXVIII. Coloring Matter," page 190. If the wool is not dyed, examine the solution spectroscopically.

If too dilute, the solution may often be concentrated by precipitating the coloring matter as a lake,<sup>d</sup> that is, allow it to settle, decant the water, dissolve in hydrochloric acid, and make alkaline with ammonium hydroxid.

In extracting with 50 per cent alcohol, the proteids of the meat are coagulated, with the formation of a pale, almost white, color. If the meat is not discolored during this extraction, it is probable that some foreign color is present.

Marpmann<sup>e</sup> examines sausages microscopically for the presence of coloring matter after dehydrating with alcohol and xylol consecutively, removing the xylol with carbon tetrachlorid, and immersing in cedar oil until the natural colors of the meat have disappeared.

<sup>a</sup> Handbuch der Fleischbeschau, 3d ed., p. 826.

<sup>b</sup> Klinger and Bujard, Zts. angew. Chem., 1891, 515.

<sup>c</sup> Spaeth, Pharm. Centralh., 1897, 38: 884.

<sup>d</sup> Bremer, Forschungs-Ber. Lebensm., 1897, 4: 45.

<sup>e</sup> Zts. angew. Mikros., 1895, 1: 12.

## MEAT EXTRACTS.

### 1. Preparation of Sample.—Provisional.

Liquid and semiliquid meat extracts and similar preparations should be removed from the container and thoroughly mixed before sampling. In many liquid preparations a sediment forms which should be carefully removed from the bottom of the container and included in the sample.

### 2. Moisture.—Provisional.

Follow the directions given on page 38, under "VI. General Methods," employing about 2 grams of powdered preparations, about 3 grams of preparations of pasty consistency, or from 5 to 10 grams of liquid extracts, according to the solid contents. Dry the powdered preparations directly without admixture. Dissolve the pasty preparations in water and dry with sufficient ignited asbestos or pumice stone to absorb the solution. Tin or lead dishes or Hofmeister glass dishes are often convenient with samples in which the residue is to be extracted for fat, as the dishes may be cut or broken and placed in the extraction tube with the sample.

### 3. Ash.—Provisional.

Proceed as directed on page 38, under "VI. General Methods." To pasty preparations add sufficient water to effect solution and evaporate to dryness in order that the solids may be distributed evenly over the bottom of the dish.

### 4. Phosphoric Acid.—Provisional.

The organic matter should be destroyed by one of the methods given on page 2, under "I. Fertilizers," and phosphoric acid determined by either the gravimetric or volumetric molybdate method described under "I. Fertilizers," on pages 3 and 4.

### 5. Chlorin.—Provisional.

Determine chlorin by Volhard's method. (Page 23, under "III. Inorganic Plant Constituents"). For ordinary purposes the solution of the ash may be employed. More exact results may be obtained by the following procedure: Dissolve about 1 gram of the meat extract in 20 cc of a 5 per cent solution of sodium carbonate, evaporate to dryness, and thoroughly ignite. Extract the residue with hot water, filter, and wash; return filter and contents to a platinum dish and ignite. Dissolve the contents of the dish in nitric acid, add the solution to the filtrate, and determine the chlorin contents as indicated above.

### 6. Fat.—Provisional.

Transfer the residue from the determination of water to a continuous-extraction apparatus, wash any fat adhering to the dish into the tube with ether, and proceed as directed on page 39, under "VI. General Methods."

### 7. Nitrogenous Substances.

#### (a) TOTAL NITROGEN.—PROVISIONAL.

Employ either the Kjeldahl or the Gunning method, page 5, under "I. Fertilizers."

(b) MEAT FIBER.<sup>a</sup>—PROVISIONAL.

Dissolve in cold water 5 grams of powdered preparations, from 8 to 10 grams of extracts of pasty consistency, or from 20 to 25 grams of fluid extracts; filter and wash with cold water. Transfer the filter paper and contents to a Kjeldahl flask and determine nitrogen as directed under total nitrogen. In case of a large amount of insoluble matter, make up to a definite volume, filter through a folded filter, and determine nitrogen in an aliquot of the filtrate; deduct the percentage of nitrogen in the total filtrate from the percentage of total nitrogen to obtain the percentage of nitrogen in meat fiber. Multiply the percentage of nitrogen by 6.25 to find the percentage of meat fiber.

## (c) COAGULABLE PROTEIDS.—PROVISIONAL.

Employ as large an aliquot of the filtrate as practicable when the nitrogen of meat fiber has been determined by difference. Make the filtrate from the meat fiber slightly acid by the addition of acetic acid or sodium hydroxid, as may be necessary; boil for two or three minutes, cool to room temperature, dilute to 500 cc, and pass through a folded filter.<sup>b</sup>

Determine nitrogen in 50 cc of the filtrate by the Kjeldahl or Gunning method (page 5, under "I. Fertilizers.").

Ten times the nitrogen so obtained deducted from the percentage of soluble nitrogen (which in turn is obtained by deducting percentage of nitrogen occurring as meat fiber from the total nitrogen) gives the percentage of nitrogen present as coagulable proteids. Multiply this figure by 6.25 for the percentage of coagulable proteids in the sample.

## (d) SYNTONIN (ACID ALBUMIN).—PROVISIONAL.

Exactly neutralize the filtrate from the determination of coagulable proteids with sodium hydroxid, using litmus as indicator, and allow to stand until the precipitate settles. If only a small amount of syntonin is precipitated, separate it with an ordinary filter, wash with water, and determine its nitrogen content by means of the Kjeldahl or Gunning method. If present in any considerable quantity, dilute to a definite volume, filter through a folded filter, and determine nitrogen in 50 cc of the filtrate.

Deduct the nitrogen thus obtained (calculated to total volume) from the nitrogen in the filtrate from the coagulable proteids (c) to obtain the syntonin nitrogen. Multiply this by 6.25 to obtain the syntonin.

## (e) AMMONIA.—PROVISIONAL.

Employ the magnesium-oxid method as directed on page 9, under "I. Fertilizers."

(f) PROTEOSES AND GELATIN.<sup>c</sup>—PROVISIONAL.

Evaporate the filtrate from the determination of syntonin to a small volume and saturate with zinc sulphate. (Use as large an aliquot of the filtrate as is practicable when the percentage of syntonin is determined by difference.) About 85 grams of powdered zinc sulphate are necessary for the saturation of

<sup>a</sup> Allen, Commercial Organic Analysis, 2d ed., 4: 324.

<sup>b</sup> It is always tedious and unsatisfactory and sometimes almost impossible to filter and wash the coagulated proteids. The work is greatly simplified, therefore, by using a folded filter and employing aliquots of the filtrate, as in this way complete filtration and washing of precipitates becomes unnecessary.

<sup>c</sup> Bömer, Zts. anal. Chem., 1895, 5: 562; also Mallet, U. S. Dept. Agr., Division of Chemistry, Bul. 54.

50 cc of the liquid at room temperature. The liquid must be saturated with the salt, but a large excess should be avoided, as it is likely to cause bumping in the subsequent determination of nitrogen. Let stand several hours, filter, and wash the precipitate with saturated zinc sulphate. In case the precipitate is voluminous, which rarely happens, make up the mixture to a definite volume with saturated zinc sulphate, filter, and determine the nitrogen in an aliquot of the filtrate; determine the nitrogen of the precipitated proteids by difference.

(g) GELATIN—STUTZER'S METHOD <sup>a</sup> MODIFIED.<sup>b</sup>—PROVISIONAL.

Boil 10 grams of the sample for a few minutes with water; filter, wash, and evaporate the filtrate to dryness in a porcelain dish of about 10 cm diameter, after the addition of about 20 grams of sand which has been freed from dust by sifting, and thoroughly ignited. Exhaust the residue with four 100 cc portions of absolute alcohol and pass the supernatant liquid through an asbestos filter, which rests on a porous plate of about 4 cm diameter, in a funnel. The funnel is surrounded by pounded ice and attached to an aspirator, by means of which gentle and gradually increasing suction may be applied. Take care to transfer as little as possible of the insoluble residue to the filter. Extract the residue repeatedly with 100 cc portions of a mixture containing 100 cc of 95 per cent alcohol, 300 grams of ice, and 600 grams of cold water, taking care that the temperature shall not exceed 5° C. Continue the extraction until the various portions of solvent used are entirely colorless. Filter the extract through the funnel employed for the alcohol extract. Finally, return the asbestos filter to the beaker which contains the exhausted residue and thoroughly extract the whole with boiling water. Receive the hot water extract in a Kjeldahl flask, determine nitrogen, and multiply the percentage of nitrogen by 5.55 to obtain the percentage of gelatin.

(h) PROTEOSES.—PROVISIONAL.

Deduct the nitrogen in the gelatin precipitate (g) from that of the proteose and gelatin precipitate. Multiply by 6.25 to obtain the percentage of proteoses.

(i) MEAT BASES.—PROVISIONAL.

Deduct from the total nitrogen (a) the sum of the nitrogen (b), (c), (d), (f), and (g). Multiply the difference by 3.12 to obtain the percentage of meat bases.

8. Glycogen.—Provisional.

Proceed as directed on page 110, under "Meat."

9. Detection of Preservatives.

Proceed as directed under "XXVII. Food Preservatives," page 179.

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<sup>a</sup> Zts. anal. Chem., 1895, 34: 568.

<sup>b</sup> U. S. Dept. of Agr., Bureau of Chemistry, Bul. 13, Part 10, p. 1397. The results obtained by this method do not include the products found by the protracted digestion of gelatin with acids or boiling water.

## **XVIII. METHODS FOR THE ANALYSIS OF DAIRY PRODUCTS.**

### **MILK.**

#### **1. Total Solids.—Official.**

##### **(a) METHOD I.**

Heat from 3 to 5 grams of milk at the temperature of boiling water until it ceases to lose weight, using a tared flat dish of not less than 5 cc diameter. If desired from 15 to 20 grams of pure dry sand may be previously placed in the dish. Cool in a desiccator and weigh rapidly to avoid absorption of hydropscopic moisture.

##### **(b) METHOD II. (BARCOCK ASBESTOS METHOD.)**

Provide a hollow cylinder of perforated sheet metal, 60 mm long and 20 mm in diameter, closed 5 mm from one end by a disk of the same material. The perforations should be about 0.7 mm in diameter and about 0.7 mm apart. Fill loosely with from 1.5 to 2.5 grams of freshly ignited, woolly asbestos, free from fine and brittle material, cool in a desiccator, and weigh. Introduce a weighed quantity of milk (between 3 and 5 grams) and dry at the temperature of boiling water to constant weight.

#### **2. Ash.—Official.**

Weigh about 20 grams of milk in a weighed dish, add 6 cc of nitric acid, evaporate to dryness, and ignite at a temperature just below redness until the ash is free from carbon.

#### **3. Total Nitrogen.—Official.**

Place about 5 grams of milk in a Kjeldahl digestion flask and proceed, without evaporation, as described under "I. Fertilizer Methods" for the determination of nitrogen, page 5. Multiply the percentage of nitrogen by 6.38 to obtain nitrogen compounds.

#### **4. Casein and Albumin in Cow's Milk.**

##### **(a) CASEIN.—OFFICIAL.**

The determination should be made when the milk is fresh, or nearly so. When it is not practicable to make this determination within twenty-four hours, add 1 part of formaldehyde to 2,500 parts of milk, and keep in a cool place. Place about 10 grams of milk in a beaker with about 90 cc of water at 40° to 42° C., and add at once 1.5 cc of a 10 per cent acetic acid solution. Stir with a

glass rod and let stand from three to five minutes longer. Then decant on filter, wash two or three times with cold water by decantation, and transfer precipitate completely to filter. Wash once or twice on filter. The filtrate should be clear, or very nearly so. If it be not clear when it first runs through, it can generally be made so by two or three repeated filtrations, after which the washing of the precipitate can be completed. Determine nitrogen in the washed precipitate and filter paper by the Kjeldahl or Gunning method ("I. Fertilizers," p. 5). To calculate the equivalent amount of casein from the nitrogen multiply by 6.38.

In working with milk which has been kept with preservatives, the acetic acid should be added in small proportions, a few drops at a time, with stirring, and the addition continued until the liquid above the precipitate becomes clear, or very nearly so.

(b) ALBUMIN.—PROVISIONAL.

Exactly neutralize with caustic alkali the filtrate obtained in the preceding operation (a), add 0.3 cc of a 10 per cent solution of acetic acid and heat the liquid to the temperature of boiling water until the albumin is completely precipitated, collect the precipitate on a filter, wash, and determine the nitrogen therein. Nitrogen multiplied by 6.38 equals albumin.

(c) CASEIN.—OPTIONAL OFFICIAL.

To 10 cc of milk add 50 cc of distilled water at 40° C., then add 2 cc of alum solution saturated at 40° C. or higher. Allow precipitate to settle, transfer to a filter, and wash. Treat the precipitate and filter paper by the Kjeldahl or Gunning method ("I. Fertilizers," p. 5).

(d) ALBUMIN.—OPTIONAL PROVISIONAL.

To the filtrate obtained from the casein determination (c) add 0.3 cc of a 10 per cent acetic-acid solution, boil the liquid until the albumin is completely precipitated and proceed as in the provisional method for albumin (b).

5. Milk Sugar. (Lactose.)

(a) OPTICAL METHOD.—OFFICIAL.

(1) PREPARATION OF REAGENTS.

(a) *Acid mercuric nitrate*.—Dissolve mercury in double its weight of nitric acid, specific gravity 1.42, and dilute with an equal volume of water. One cubic centimeter of this reagent is sufficient for the quantities of milk mentioned below. Larger quantities may be used without affecting the results of polarization.

(b) *Mercuric iodid with acetic acid*.—Mix 33.2 grams of potassium iodid, 13.5 grams of mercuric chlorid, 20 cc of glacial acetic acid, and 640 cc of water.

(2) DETERMINATION.

The milk should be at a constant temperature, and its specific gravity determined with a delicate hydrometer. When greater accuracy is required, a pycnometer is used.

The quantities of the milk measured for polarization vary with the specific gravity of the milk as well as with the polariscope used. The quantity to be measured in any case will be found in the following table;

*Determination of volume of milk sample.*

Specific gravity.	Volume of milk to be used.	
	For polariscopes of which the sucrose normal weight is 16.19 grams.	For polariscopes of which the sucrose normal weight is 26.048 grams.
1.024	cc 60.0	cc 64.4
1.026	59.9	64.3
1.028	59.8	64.15
1.030	59.7	64.0
1.032	59.6	63.9
1.034	59.5	63.8
1.035	59.35	63.7

Place the quantity of milk indicated in the table in a flask graduated at 102.4 cc for a Laurent or 102.6 cc for a Ventzke polariscope (Mohr cc). Add 1 cc of mercuric nitrate solution or 30 cc of mercuric iodid solution (an excess of these reagents does no harm), fill to the mark, agitate, filter through a dry filter, and polarize. It is not necessary to heat before polarizing. In case a 200 mm tube is used, divide the polariscope reading by 3 when the sucrose normal weight for the instrument is 16.19 grams, or by 2 when the normal weight for the instrument is 26.048. When a 400 mm tube is used, these divisors become 6 and 4, respectively. For the calculation of the above table the specific rotary power of lactose is taken as  $52.53^\circ$ , and the corresponding number for sucrose as  $66.5^\circ$ . The lactose normal weight to read  $100^\circ$  on the sugar scale for Laurent instruments is 20.496 grams, and for Ventzke instruments, 32.975 grams. In case metric flasks are used the weights here mentioned must be reduced to 16.160 and 26.000 grams, respectively.

## (b) GRAVIMETRIC METHOD.—OFFICIAL.

## (1) PREPARATION OF THE MILK SOLUTION.

Dilute 25 cc of the milk with 400 cc of water and add 10 cc of a solution of copper sulphate of the strength given for Soxhlet's modification of Fehling's solution, page 42, "VI. General Methods;" add about 7.5 cc of a solution of potassium hydroxid of such strength that one volume of it is just sufficient to completely precipitate the copper as hydroxid from one volume of the solution of copper sulphate. Instead of a solution of potassium hydroxid of this strength 8.8 cc of a half-normal solution of sodium hydroxid may be used. After the addition of the alkali solution the mixture must still have an acid reaction and contain copper in solution. Fill the flask to the 500 cc mark, mix, and filter through a dry filter.

## (2) DETERMINATION.

Proceed as directed under "VI. General Methods," page 48.

## 6. Fat.

## (a) BABCOCK ASBESTOS METHOD.—OFFICIAL.

Extract the residue from the determination of water by the Babcock asbestos method with anhydrous ether until all the fat is removed, evaporate the ether, dry the fat at the temperature of boiling water, and weigh. The fat may also be determined by difference, drying the extracted cylinders at the temperature of boiling water.

## (b) PAPER COIL METHOD.—OFFICIAL.

Make coils of thick filter paper, cut into strips 6.25 by 62.5 cm, and thoroughly extract with ether and alcohol, or correct the weight of the extract by a constant obtained for the paper. From a weighing bottle or weighing pipette transfer about 5 grams of milk to the coil, care being taken to keep the end of the coil held in the fingers dry. Dry the coil, dry end down, on a piece of glass at the temperature of boiling water for one hour, or, better, in hydrogen at the temperature of boiling water; transfer to an extraction apparatus, and extract with absolute ether or petroleum ether boiling at about 45° C.; dry the extracted fat and weigh.

## (c) VOLUMETRIC METHODS.—OFFICIAL.

Babcock's or Gerber's centrifugal methods may be used. Following is the Babcock centrifugal method:

## (1) APPARATUS.

(a) *Babcock milk-test bottles*, graduated to 10 per cent.

(b) *A centrifuge* with sockets for from 2 to 32 bottles, according to the number of tests to be made, and capable of being run at a speed of from 600 to 1,200 revolutions per minute, according to the diameter of the machine. If many tests are made steam turbine testers or electrical testers will be found convenient.

(c) *Pipettes*, 17.6 cc.

(d) *Graduates*, 17.5 cc, or a Swedish acid bottle delivering that amount, for measuring sulphuric acid.

## (2) DETERMINATION.

Pipette off 17.6 cc of the carefully mixed sample into a test bottle and add 17.5 cc of commercial sulphuric acid (specific gravity, 1.82–1.83). Mix, and when the curd is dissolved whirl the test bottles in the centrifuge for four minutes at the required speed for the machine used. Add boiling hot water, filling to the neck of the bottles, and whirl for one minute; again add boiling water so as to bring the fat within the scale on the neck of the bottles, and after whirling for one minute more read the length of the fat column, care being taken to make the readings at a temperature between 130° and 150° F. when the fat is wholly liquid. The readings give the per cent of fat in the milk direct.

For details as to the manipulation of the Babcock test and its application in the analysis of dairy products other than milk the following books may be consulted: Farrington and Woll, *Testing Milk and Its Products*, 18th edition, 1908, Madison, Wis., and Van Slyke, *Modern Methods of Milk Testing*, 1906, New York.

## 7. Detection of Added Water. Zeiss Immersion Refractometer Method.—Provisional.

To 100 cc of milk at a temperature of about 20° C. add 2 cc of 25 per cent acetic acid (sp. gr. 1.035) in a beaker; and heat the beaker, covered with a watch glass, in a water bath for twenty minutes at a temperature of 70° C. Place the beaker in ice water for ten minutes and separate the curd from the serum by filtering through a 12.5 cm folded filter. Transfer about 35 cc of the serum to one of the beakers that accompanies the control-temperature bath used in connection with the Zeiss immersion refractometer, and take the refractometer reading at exactly 20° C., using a thermometer graduated to tenths of a degree. A reading below 39 indicates added water; between 39 and 40 the sample is suspicious.



### 8. Detection of Gelatin.—Provisional.

Prepare an acid solution of mercuric nitrate by dissolving mercury in twice its weight of nitric acid of 1.42 specific gravity, and diluting this solution to 25 times its bulk with water. To 10 cc of the milk or cream to be examined, add an equal volume of the acid mercuric nitrate solution, shake the mixture, add 20 cc of water, shake again, allow to stand five minutes, and filter. If much gelatin is present the filtrate will be opalescent and can not be obtained quite clear. To a portion of the filtrate contained in a test tube, add an equal volume of a saturated aqueous solution of picric acid. A yellow precipitate will be produced in presence of any considerable amount of gelatin, while smaller amounts will be indicated by a cloudiness. In the absence of gelatin the filtrate obtained will remain perfectly clear.

### 9. Detection of Formaldehyde.—Provisional.

Use the method described on page 183, under "XXVII. Food Preservatives."

### 10. Detection of Borax and Boric Acid.—Provisional.

Use the method described on page 183, under "XXVII. Food Preservatives."

### 11. Detection of Benzoic Acid.—Provisional.

Add 5 cc of dilute hydrochloric acid to 50 cc of the milk in a flask and shake to curdle. Then add 150 cc of ether, cork the flask and shake well. Break up the emulsion which forms by aid of a centrifuge, or if the latter is not available extract the curdled milk by gently shaking with successive portions of ether, avoiding the formation of an emulsion. Transfer the ether extract (evaporated to small volume if large in bulk) to a separatory funnel and separate the benzoic acid from the fat by shaking out with dilute ammonium hydroxid, which takes out the former as ammonium benzoate. Evaporate the ammoniacal solution in a dish over the water bath till all free ammonia has disappeared, but before dryness is reached add a few drops of ferric chlorid reagent. The characteristic flesh-colored precipitate indicates benzoic acid. Care should be taken not to add the ferric chlorid until all the ammonia has been driven off, otherwise a precipitate of ferric hydrate is formed. (See also "XXVII. Food Preservatives," p. 181.)

### 12. Detection of Salicylic Acid.—Provisional.

Proceed exactly as directed for benzoic acid in the preceding section. On applying the ferric chlorid to the solution after evaporation of the ammonia the well-known violet color indicates salicylic acid.

### 13. Detection of Foreign Color.

#### (a) LEACH'S METHOD.—PROVISIONAL.

Warm about 150 cc of milk in a casserole over the flame and add about 5 cc of acetic acid, after which slowly continue the heating nearly to the boiling point while stirring. Gather the curd, when possible, into one mass by the stirring rod, and pour off the whey. If the curd breaks up into small flecks separate from the whey by straining through a sieve or colander. Press the

curd free from adhering liquid, transfer to a small flask, and macerate for several hours (preferably overnight) in about 50 cc of ether, the flask being tightly corked and shaken at intervals.

(1) DETECTION OF ANNATTO (IN THE ETHER EXTRACT).

Decant the ether extract as obtained above into an evaporating dish, place on the water bath, and evaporate the ether. Make the fatty residue alkaline with sodium hydroxid, and pour upon a very small wet filter while still warm. After the solution has passed through, wash the fat from the filter with a stream of water and dry the paper. If, after drying, the paper is colored orange, the presence of annatto is indicated. Confirm by applying a drop of stannous chlorid solution, which, in presence of annatto, produces a characteristic pink on the orange-colored paper.

(2) DETECTION OF ANILIN ORANGE (IN THE CURD).

The curd of an uncolored milk is perfectly white after complete extraction with ether, as is also that of a milk colored with annatto.

If the extracted fat-free curd is distinctly dyed an orange or yellowish color, anilin orange is indicated. To confirm the presence of this color, treat a lump of the fat-free curd in a test tube with a little strong hydrochloric acid. If the curd immediately turns pink, the presence of anilin orange is assured.

(3) DETECTION OF CARAMEL (IN THE CURD).

If the fat-free curd is colored a dull brown, caramel is to be suspected. Shake a lump of the curd, as in (2), with strong hydrochloric acid in a test tube and heat gently. In the presence of caramel the acid solution will gradually turn a deep blue, as will also the white, fat-free curd of an uncolored milk, while the curd itself does not change color. It is only when this blue coloration of the acid occurs in connection with a brown colored curd, which itself does not change color, that caramel is to be suspected, as distinguished from the pink coloration produced at once under similar conditions by anilin orange.

(b) LYTNGOE'S TEST FOR ANILIN ORANGE.—PROVISIONAL.

Treat about 10 cc of the milk with an equal volume of hydrochloric acid (sp. gr. 1.20) in a porcelain casserole and give the dish a slight rotary motion. If an appreciable amount of anilin orange is present, a pink color will at once be imparted to the curd particles as they separate.

### CREAM.

Most of the methods for milk analysis may be applied to cream, properly diluted. For accurate work the dilution should in most cases be made after the cream has been weighed (not measured) for the determination, since cream tends to separate quickly from water. In the Babcock centrifugal method the cream should always be weighed into the test bottle before diluting.

### CONDENSED MILK.—PROVISIONAL.

#### 1. Preparation of Sample.

Mix thoroughly by transferring the contents of the can to a large evaporating dish and stirring it with a pestle until homogeneous. Weigh 40 grams of the mixed sample in a 100 cc flask, or transfer thereto by washing, and make up to the mark with water.

## 2. Total Solids.

Dilute a measured portion of the above 40 per cent solution with an equal amount of water, use 5 cc of the diluted mixture, corresponding to 1 gram of the condensed milk, and proceed as directed under "Milk," page 117.

## 3. Ash.

Ignite the total solids at very low redness, cool, and weigh.

## 4. Proteids.

Determine nitrogen according to the Kjeldahl or Gunning method ("I. Fertilizers," p. 5) in 5 cc of the 40 per cent solution and multiply by 6.38.

## 5. Lactose.

Dilute 5 cc of the 40 per cent solution to about 40 cc and add 0.6 cc of Fehling's copper solution. Nearly neutralize with sodium hydroxid, make up to 100 cc, filter through a dry filter, and determine lactose in an aliquot as directed under "VI. General Methods," page 48.

## 6. Sucrose.

Determine by difference, deducting the milk solids (lactose+proteids+fat+ash) from the total solids.

## 7. Fat or Ether Extract.

(See also Appendix, p. 253.)

Place 15 cc of the 40 per cent solution in a Babcock test bottle. Fill the bottle nearly to the neck with water, add 4 cc of Fehling's copper solution, shake thoroughly and rapidly, separating the precipitated proteids and fat by means of a centrifuge,<sup>a</sup> or the precipitate may be allowed to settle, which it does more quickly in the cold. Withdraw the supernatant liquid by means of a small-stemmed pipette with a wisp of wet absorbent cotton twisted over the bottom to serve as a filter. Wipe off the cotton into the bottle on withdrawing the pipette. Give the precipitated proteids and fat two additional washings by shaking with water, separating the precipitate, and removing the washings with the pipette. If the precipitate is caked hard after centrifuging, use a stiff platinum wire as a stirrer. Finally, add water to an approximate volume of 17.5 cc and 17.5 cc of sulphuric acid, and continue the test as in the Babcock process of milk testing, multiplying the reading by 3 for the percentage of fat in the sample.

# BUTTER AND ITS SUBSTITUTES.

## 1. Preparation of Sample.—Official.

If large quantities of butter are to be sampled, use a butter trier or sampler. Completely melt the portions thus drawn, 100 to 500 grams, in a closed vessel at as low a temperature as possible. When melted, cool the whole, and at the same time shake the mass violently until it is homogeneous and sufficiently solidified to prevent the separation of the water and fat. Then pour a portion into the vessel from which it is to be weighed for analysis. The sample should completely or nearly fill the vessel and should be kept in a cold place until analyzed.

<sup>a</sup> While the steam-driven centrifuge may be used, it is better to centrifuge in the cold, since the heat of the steam-driven machine cakes the precipitate so that it is harder to wash.

## 2. Moisture.—Official.

Place 1.5 to 2.5 grams in a dish with a flat bottom having a surface of at least 20 sq. cm. and dry at the temperature of boiling water until it ceases to lose weight, each drying to be for only one hour. The use of clean dry sand or asbestos is admissible and is necessary if a dish with a round bottom be employed.

## 3. Casein, Ash, and Chlorin.—Official.

Cover the crucible containing the residue from the fat determination by the indirect method (see 4 (a)) and heat gently at first, gradually raising the temperature to just below redness. The cover may then be removed and the heat continued until the contents of the crucible are white. The loss in weight represents casein, and the residue in the crucible, mineral matter. In this mineral matter, dissolved in water slightly acidulated with nitric acid, determine chlorin, either gravimetrically or volumetrically.

## 4. Ether Extract.

### (a) INDIRECT METHOD.—OFFICIAL.

Dissolve in the dish with absolute ether or petroleum ether the dry butter obtained in the water determination in which no absorbent was used, transfer to a weighed gouch with the aid of a wash bottle filled with the solvent and wash until free from fat. Dry the crucible and contents at the temperature of boiling water until the weight is constant and calculate the fat.

### (b) DIRECT METHOD.—OFFICIAL.

From the dry butter obtained in determining the water, either with or without the use of an absorbent, extract the fat with anhydrous alcohol-free ether, receiving the solution in a weighed flask. Evaporate the ether and dry the extract at the temperature of boiling water until it ceases to lose weight, the dryings not to exceed one hour each in duration.

## 5. Salt.—Official.

Weigh in a counterpoised beaker from 5 to 10 grams of butter, using portions of about 1 gram from different parts of the sample. Add about 20 cc of hot water and after the butter is melted transfer the whole to a separatory funnel. Insert the stopper and shake for a few moments. Let stand until the fat has all collected on the top of the water, then draw off the latter into a flask, being careful to let none of the fat globules pass. Again add hot water to the beaker and repeat the extraction from ten to fifteen times, using each time from 10 to 20 cc of water. The washings will contain all but a mere trace of the sodium chlorid originally present in the butter. Determine its amount in the whole or an aliquot of the liquid by the volumetric silver-nitrate method, with potassium chromate as indicator.

## 6. Examination of Fat.

### (a) PREPARATION OF SAMPLE.—OFFICIAL.

Melt the butter and keep it in a dry place at about 60° C. for two or three hours, until the water and curd have entirely separated. Pour off the clear supernatant fat and filter through a dry filter paper in a hot-water funnel containing boiling water, or in an oven at about 60° C. Should the filtered liquid fat not be perfectly clear it must be filtered again.

**(b) METHODS OF EXAMINATION OF BUTTER FAT.**

See methods under "XIX. Edible Fats and Oils," page 129.

**7. Qualitative Tests of Butter.****(a) MICROSCOPIC EXAMINATION.—OFFICIAL.**

Place a small portion of the fresh unmelted sample, taken from the inside of the mass, on a slide, add a drop of pure sweet oil, cover with gentle pressure, and examine with a one-half to one-eighth inch objective for crystals of lard, etc. Examine the same specimen with polarized light and a selenite plate without the use of oil. Pure fresh butter will neither show crystals nor a particolored field with selenite. Other fats melted and cooled and mixed with butter will usually present crystals and variegated colors with the selenite plate.

For further microscopic study dissolve from 3 to 4 cc of the fat in 15 cc of ether in a test tube. Close the tube with a loose plug of cotton wool and allow to stand from twelve to twenty-four hours at 20° to 25° C. When crystals form at the bottom of the tube, they are removed with a pipette, glass rod, or tube, placed on a slide, covered, and examined. The crystals formed by later deposits may be examined in a similar way.

**(b) SPECIAL TESTS FOR RENOVATED (PROCESS) BUTTER AND OLEO.****(1) FOAM TEST.—PROVISIONAL.**

Heat 2 or 3 grams of the sample, either in a spoon or dish, over a free flame. True butter will foam abundantly, whereas process butter will bump and sputter, like hot grease, without foaming. Oleo behaves like process butter, but chemical tests will determine whether the sample is oleomargarine or butter.

**(2) APPEARANCE OF THE MELTED FAT.—PROVISIONAL.**

Melt from 50 to 100 grams of butter or process butter at 50° C. The curd from butter will settle, leaving a clear supernatant fat. On the other hand, the supernatant fat in the case of process butter does not assume that clear appearance, but remains more or less turbid.

**(3) MICROSCOPIC EXAMINATION.—PROVISIONAL.**

Place a bit of the butter or process butter on a glass slide, cover it and press it into a thin film with cover glass. Examine immediately with a polarizing microscope magnifying from 100 to 140 diameters. When a selenite plate is placed between the slide and the lower nicol a normal butter will give a uniformly blue colored field, showing the absence of fat crystals.

**(c) DETECTION OF BORIC ACID.—PROVISIONAL.**

Melt about 25 grams of the sample on the water bath, pour off the fat from the aqueous solution that settles, slightly acidify the aqueous solution with hydrochloric acid, and test in the usual manner with turmeric paper for boric acid. (See p. 183, under "XXVII. Food Preservatives.")

**(d) DETECTION OF ANNATTO AND SAFFRON.****CORNWALL'S METHOD.—PROVISIONAL.**

Dissolve 5 grams of the fat in 50 cc of ether in a wide tube and shake the solution vigorously with 12 to 15 cc of a very dilute solution of potassium hydroxid, which must still be alkaline after it separates from the ether solution.

Allow to stand a few hours, then draw off the aqueous layer, evaporate to dryness, and test with sulphuric acid which in the presence of annatto gives first a blue or violet blue, changing quickly to green and finally to brown.

Saffron, which would be extracted at the same time, acts differently when treated with sulphuric acid, not giving the green coloration.

The aqueous solutions, if not clear enough to use, must not be filtered, as the filter paper will take up large amounts of the color, but can be shaken up again with fresh portions of ether. Carbon disulphid may also be used as a solvent. Uncolored butters treated in this way give only a very slight trace of coloring matter.

(2) MASSACHUSETTS BOARD OF HEALTH METHOD FOR ANNATTO.—PROVISIONAL.

Treat 2 or 3 grams of the melted and filtered fat (freed from salt and water) with warm dilute sodium hydroxid, and after stirring pour the mixture while warm upon a wet filter, using to advantage a hot funnel. If annatto is present the filter will absorb the color so that when the fat is washed off by a gentle stream of water the paper will be dyed straw color. It is well to pass the warm, alkaline filtrate two or three times through the fat on the filter to insure removal of the color. If, after drying the filter, the color turns pink on application of a drop of stannous chlorid solution the presence of annatto is assured.

(3) GEISLER'S METHOD FOR AZO COLORS.—PROVISIONAL.

Spread a few drops of the clarified fat upon a porcelain surface and add a pinch of fuller's earth. In the presence of various azo dyes a pink to violet red coloration will be produced in a few minutes. Some varieties of the fuller's earth react much more readily than others with azo colors.

(4) LOW'S METHOD FOR AZO COLORS.—PROVISIONAL.

Melt a small amount of the fat in a test tube, add an equal volume of the mixture of one part of concentrated sulphuric acid and four parts of glacial acetic acid and heat nearly to the boiling point, the liquids being thoroughly mixed by shaking. Then set aside and after the acid solution has settled it will be colored wine red in the presence of azo color, while with pure butter fat comparatively no color will be produced.

(5) ACID AND ALKALI TESTS.—PROVISIONAL.

Pour into each of two test tubes about 2 grams of the filtered fat dissolved in ether. Into one of the tubes pour 1 or 2 cc of hydrochloric acid and into the other about the same volume of dilute potassium hydroxid solution. Shake the tubes well and allow to stand. In the presence of azo dye the test tube to which the acid has been added will show a pink to wine-red coloration, while the potash solution in the other tube will show no color. If, on the other hand, annatto or other vegetable color has been used the potash solution will be colored yellow, while no color will be apparent in the acid solution.

## CHEESE.

### 1. Selection and Preparation of Sample.—Official.

When the cheese can be cut take a narrow wedge-shaped segment reaching from the outer edge to the center of the cheese. Cut this into strips and pass through a sausage grinding machine three times. When the cheese can not be

cut take the sample with a cheese trier. If only one plug can be obtained take it perpendicular to the surface of the cheese at a point one-third of the distance from the edge to the center and extending either entirely or half way through it. When possible draw three plugs—one from the center, one from a point near the outer edge, and one from a point half way between the other two. For inspection purposes reject the rind, but for investigations requiring the absolute amount of fat in the cheese include the rind in the sample. It is preferable to grind the plugs in a sausage machine, but when this is not done they are cut very fine and carefully mixed.

## 2. Moisture.—Official.

Place from 2 to 5 grams of cheese in a weighed platinum or porcelain dish which contains a small quantity of porous material, such as ignited asbestos or sand, to absorb the fat which may run out of the cheese. Heat in a water oven for ten hours and weigh; the loss in weight is considered as moisture. Or, if preferred, the dish may be placed in a desiccator over concentrated sulphuric acid and dried to constant weight. In some cases this may require as much as two months. The acid should be renewed when the cheese has become nearly dry.

## 3. Ash.—Official.

The dry residue from the moisture determination may be used for the ash. If the cheese be rich in fat, the asbestos will be saturated therewith. Ignite cautiously to avoid spurting, removing the lamp while the fat is burning off. When the flame has died out, the burning may be completed in a muffle at low redness. When desired, the salt may be determined in the ash in the manner specified under butter analysis, page 124.

## 4. Nitrogen.—Official.

Determine nitrogen by the Kjeldahl or Gunning method, as given on page 5, under "I. Fertilizers," using about 2 grams of cheese, and multiply the percentage of nitrogen by 6.38 to obtain the nitrogen compounds.

## 5. Fat.

### (a) GRAVIMETRIC METHOD.—OFFICIAL.

Cover the perforations in the bottom of the extraction tube with dry asbestos felt, and on this place a mixture containing equal parts of anhydrous copper sulphate and pure, dry sand to the depth of about 5 cm, packing loosely. Cover the upper surface of this material with a film of asbestos. Place on this 2 to 5 grams of the sample and extract with anhydrous ether for five hours in a continuous extraction apparatus. Remove the cheese and grind it to a fine powder with pure sand in a mortar, replace the mixed cheese and sand in the extraction tube, washing the mortar with ether and adding the washings to the tube, and continue the extraction for at least ten hours.

### (b) BABCOCK CENTRIFUGAL METHOD.—PROVISIONAL.

Weigh about 6 grams of cheese in a tared dish. Add 10 cc of boiling water and stir with a rod until the cheese softens and an even emulsion is formed, preferably adding a few drops of strong ammonium hydroxid, and keep the beaker in hot water until the emulsion is nearly completed and the mass free from lumps.

If the sample is a full cream cheese a Babcock cream bottle is employed.

The contents of the beaker, after cooling, are transferred to the test bottle as follows: Add to the beaker about half of the 17.6 cc of sulphuric acid usually employed in this test, stir with a rod, and pour carefully into the bottle, using the remainder of the acid in two portions for washing out the beaker. Finally proceed as in the Babcock test for milk. Multiply the fat reading by 18 and divide by the weight of the sample to obtain the per cent of fat.

#### 6. Acidity.—Provisional.

To 10 grams of finely divided cheese add water at the temperature of 40° C. until the volume equals 105 cc; agitate vigorously and filter. Titrate 25 cc portions of the filtrate with standard sodium hydroxid, preferably tenth-normal, using phenolphthalein as indicator. Express amount of acid as lactic.

#### 7. Separation of Fat for Examination.—Provisional.

##### (a) METHOD I—ALKALINE EXTRACTION METHOD.

Cut about 300 grams of the cheese into fragments the size of a pea. Treat with 700 cc of potassium hydroxid (50 grams per liter) at 20° C. in a large wide-necked flask, and promote the solution of casein by vigorous shaking. In from five to ten minutes the casein will be dissolved and the fat will come to the surface in lumps. Collect the lumps of fat into as large a mass as possible by gently shaking. Pour cold water into the flask until the fat is driven up into the neck and remove it by means of a spoon. Wash the fat thus obtained with as little water as will remove the residue of the lye which it may contain. Experience has shown that the fat is not perceptibly attacked by the lye in this treatment. By this method the fat is practically all separated in a short time and is then easily prepared for chemical analysis by filtering and drying as directed in the official method, page 124.

##### (b) METHOD II—ACID EXTRACTION METHOD.

Grind the cheese by passing it through a meat-cutting machine. Transfer it to a large flask and pour warm water upon it using 1 cc for every gram of cheese. Shake thoroughly and add sulphuric acid (sp. gr. 1.82 to 1.825) slowly and in small quantities, shaking after each addition of acid. The total amount of acid used should be the same as the amount of water used. Remove the fat, which separates after standing a few minutes, by means of a separatory funnel, wash it free from acid, filter, and dry.

#### 8. Examination of the Fat.

Proceed as directed under "XIX. Edible Oils and Fats," page 129.



## XIX. METHODS FOR THE ANALYSIS OF EDIBLE FATS AND OILS.

### 1. Preparation of Sample.—Provisional.

Melt the solid fats and filter by means of a hot-water funnel or similar apparatus. Make the different determinations on samples of this melted homogeneous mass. Filter oils that are not clear. Oil and fat should always be kept in a cool place, otherwise they will soon become rancid, which will affect more or less the physical and chemical constants.

### 2. Specific Gravity.

#### (a) DETERMINATION AT 15.5° C.—PROVISIONAL.

Determine the specific gravity of oils at 15.5° C., by the use of a pycnometer Westphal balance,<sup>a</sup> or accurately graduated hydrometer.<sup>b</sup>

If determined at room temperature, the following formula may be used to calculate the specific gravity at 15.5° C.:<sup>c</sup>

$$G = G' + 0.00064 (T - 15.5^{\circ} \text{ C.}).$$

$$G = \text{sp. gr. at } 15.5^{\circ}.$$

$$G' = \text{sp. gr. at } T.$$

$$0.00064 = \text{mean correction for } 1^{\circ} \text{ C.}$$

This is only approximately correct, as the correction varies for different oils, but will satisfy ordinary requirements. If a higher degree of accuracy be desired, the factors given in the following table may be employed, but to obtain the best results the determination must be made at standard temperature.

*Factors for calculating specific gravity of oils.<sup>d</sup>*

Oil.	Correc- tion for 1° C.	Observer.	Oil.	Correc- tion for 1° C.	Observer.
Cod-liver oil.....	0.000646	A. H. Allen.	Rape oil .....	0.000620	A. H. Allen.
Lard oil .....	.000658	C. M. Wetherill.	Sesame oil .....	.000624	Do.
Olive oil.....	.000629	C. M. Stillwell.	Cotton-seed oil ....	.000629	Do.
Arachis oil.....	.000655	A. H. Allen.	Cocoonut olein.....	.000665	Do.

The following table gives correction for solid fats:

*Factors for calculating specific gravity of solid fats.<sup>e</sup>*

Fats.	Correction for 1° C.	Fats.	Correction for 1° C.
Cocoa butter.....	0.000717	Cocoonut stearin.....	0.000674
Tallow.....	.000675	Cocoonut oil.....	.000642
Lard.....	.000650	Palm-nut oil.....	.000657
Butter fat.....	.000617		

<sup>a</sup> Crampton, U. S. Dept. Agr., Division of Chemistry, Bul.-13, Part 4, p. 438.

<sup>b</sup> Accurately made hydrometers reading from 0.900 to 0.940 sp. gr. at 15.5° C., will satisfy every requirement of accuracy and speed.

<sup>c</sup> Allen, Commercial Organic Analysis, 3d. ed., 2 (1) : 33; Winton, Conn. Agr. Exper. Stat. Rept., 1900, Part 2, p. 149.

<sup>d</sup> Allen, Commercial Organic Analysis, 3d ed., 2 (1) : 33.

<sup>e</sup> Ibid., p. 32.

## (b) DETERMINATION AT 100° C.—OFFICIAL.

## (1) STANDARDIZATION OF FLASKS.

(a) *Method I.*—Use a small specific-gravity flask of from 25 to 30 cc capacity. Wash the flask thoroughly with hot water, alcohol, and ether, and then dry it. After cooling in a desiccator accurately determine the weight of the flask and stopper. Fill the flask with freshly boiled, hot, distilled water. Keep the water of the bath in brisk ebullition for thirty minutes, any evaporation from the flask being replaced by the addition of boiling distilled water. Then insert the stopper, previously heated to 100° C., remove the flask, wipe it dry, and after it has nearly cooled to room temperature place it in the balance and weigh when the balance temperature is reached.

(b) *Method II.*—The following formula may be used for calculating the weight of water ( $W^r$ ) which a given flask will hold at  $T^\circ$  (weighed in air with brass weights at the temperature of the room) from the weight of water ( $W^t$ ) (weighed in air with brass weights at the temperature of the room) contained therein at  $t^\circ$ :

$$W^r = W^t \frac{d^r}{d^t} - [1 + y(T - t)]$$

$d^r$  = the density of water at  $T^\circ$ .

$d^t$  = the density of water at  $t^\circ$ .

$y$  = the coefficient of cubical expansion of glass.<sup>a</sup>

## (2) DETERMINATION.

Rinse the flask with alcohol and ether and dry for a few minutes at the temperature of boiling water. Fill the flask with the dry, hot, fresh-filtered fat, which should be entirely free from air bubbles; replace it in the water bath, and keep for thirty minutes at the temperature of boiling water. Insert the stopper, previously heated to 100° C., remove the flask, wipe dry, place in the balance after it has nearly cooled to room temperature, and weigh when the balance temperature is reached. The weight of fat having been determined, obtain the specific gravity by dividing the weight of fat by the weight of water previously found. Example:

	Grams.
Weight of flask, dry.....	10.0197
Weight of flask, plus water.....	37.3412
Weight of water.....	27.3215
Weight of flask, plus fat.....	34.6111
Weight of fat.....	24.5914
Specific gravity = $24.5914 \div 27.3215 = 0.90008$ .	

The weight of the dry empty flask may be used constantly if great care be taken in handling and cleaning the apparatus, but the weight of water at boiling temperature must be determined under the barometric conditions prevailing at the time the determination is made.

<sup>a</sup> This factor is commonly given as 0.00026, but it varies considerably. Schulze (Zts. anal. Chem., 1882, 21: 167) found that the glass he used varied from 0.0000288 to 0.0000305—an average of 0.0000296. Ewell has used 0.000028 in his work (U. S. Dept. Agr., Division of Chemistry, Bul. 62, p. 121).

## 3. Index of Refraction.

## (a) GENERAL DISCUSSION.—PROVISIONAL.

Determine the index of refraction with any standard instrument, oils being read at 15.5° C. and fats at 40° C.

The temperature must be controlled with great care, and in accurate work the readings should be taken at standard temperature. The readings of the Zeiss butyro-refractometer can be reduced to standard temperature by the following formula: <sup>a</sup>

$$R = R' + 0.55 (T' - T),$$

in which R is the reading reduced to T, R' the reading at temperature T'. T the standard temperature, and 0.55 the correction for 1° C. in scale divisions. With oils the factor 0.58 is substituted in the formula for 0.55, since they have a higher index of refraction.

To calculate to standard temperature the readings of the instruments which give the index of refraction directly the factor 0.000365 should be used. As the temperature rises the refractive index falls. Example: The refractive index of a butter fat determined at 32.4° C.=1.4540 is reduced to 25° C., as follows: 32.4-25=7.4; 0.000365 × 7.4=0.0027; it is then 1.4540+0.0027=1.4567.

The instrument used may be set with distilled water at 18° C., the theoretical refractive index of water at that temperature being 1.3330. In the determination above given the refractive index of pure water measured 1.3300; hence the above numbers should be corrected for theory by the addition of 0.0030, making the corrected index of the butter fat mentioned at the temperature given 1.4597.

The index of refraction varies greatly with the specific gravity, increasing as it increases. In abnormal results it is often well to see if the specific refractive power <sup>b</sup> is different from the normal. Calculate the specific refrac-

tive power from the formula  $\frac{N-1}{D}$ , in which N equals the refractive index and

D the specific gravity.<sup>c</sup>

<sup>a</sup> Willey, Principles and Practice of Agricultural Analysis, 3: 341; Winton, Conn. Agr. Exper. Stat. Rept., 1900, Part 2, p. 142.

<sup>b</sup> Landolt, Ber. d. chem. Ges., 1882, 15: 1031; C. A. Browne, jr., J. Amer. Chem. Soc., 1899, 21: 991.

<sup>c</sup> Procter (J. Soc. Chem. Ind., 1898, 17: 1021) has shown that the Lorenz formula

$\frac{N^2-1}{(N^2+2)D}$  gives much more satisfactory results than  $\frac{N-1}{D}$  and gives a calculation table.

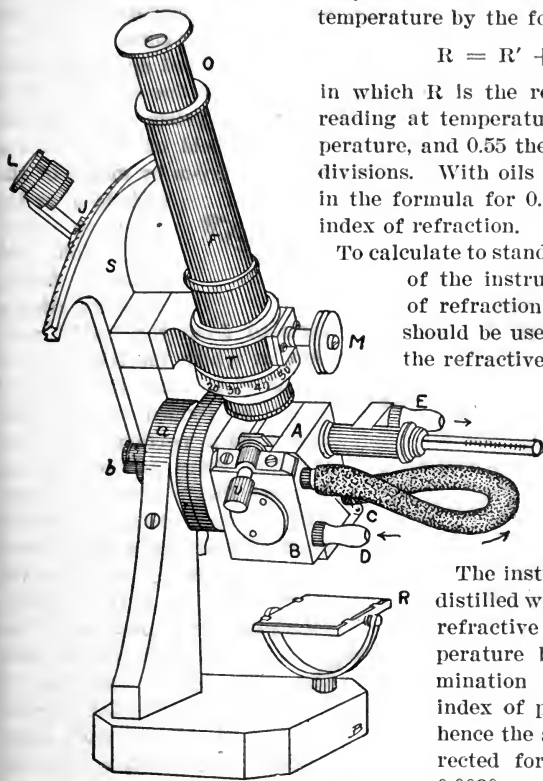


FIG. 4.—The Abbe refractometer, with temperature-controlled prisms.

## (b) ABBE'S REFRACTOMETER.—OFFICIAL.

The refractive index is conveniently determined by the Abbe refractometer, the latest model of which, with water-jacketed prisms, is shown in fig. 4. To charge the instrument open the double prism AB by means of the screw head v and place a few drops of the liquid on the prism, or, if preferred, loosen the set screw and pour a few drops of the liquid into the funnel-shaped aperture between the prisms. Then close the prisms firmly by tightening the screw head.

Allow the instrument to stand for a few minutes before the reading is made, so that the temperature of the liquid and the instrument will be the same.

The method of measurement is based upon the observation of the position of the *border line of total reflection* in relation to the faces of a prism of flint glass. Bring this border line into the field of vision of the telescope by rotating the double prism by means of the

alidade J in the following manner: Hold the sector S firmly, move the alidade

forward from the initial position, at which the index points to  $n_D=1.3$ , until the field of vision is divided into a light and a dark portion. The line dividing these portions is the "border line." This, as a rule, will not be a sharp line, but a band of color for which correction is made by rotating the screw head M until a sharp, colorless line is obtained.

The border line should now be adjusted so that it falls on the point of intersection of the two cross hairs. Read the refractive index of the substance directly on the scale of the sector. Check the correctness of the instrument

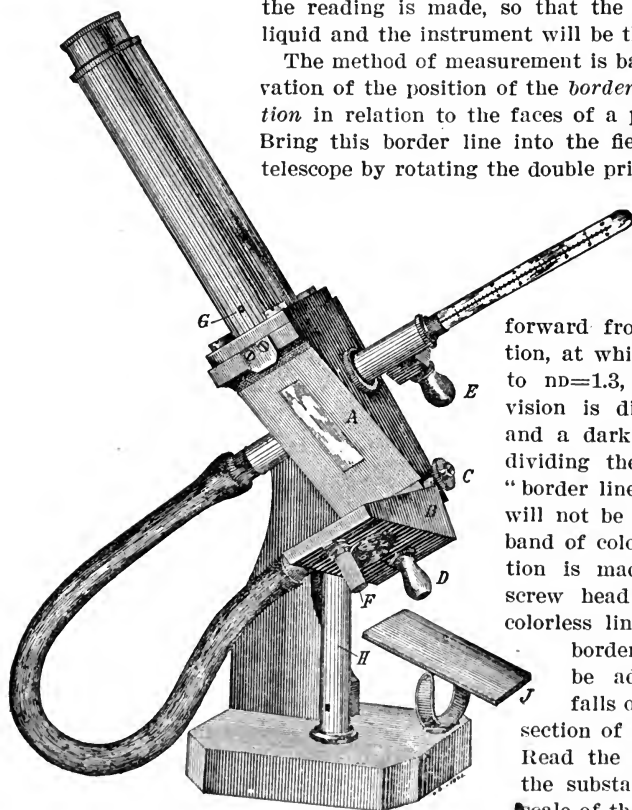


FIG. 5.—Zeiss butyro-refractometer.

by means of the quartz plate which accompanies it, using monobromonaphthalene and make the necessary correction in the reading.

(c) ZEISS BUTYRO-REFRACTOMETER.<sup>a</sup>—PROVISIONAL.

Place the instrument (fig. 5) in such a position that diffuse daylight or any form of artificial light can be readily obtained for illumination. Supply through nozzle D a stream of water of constant temperature. Then open the prism casing by giving to the pin F a half turn and carefully clean the surface of the prism. Melt the fat and filter, allowing the first two or three drops to fall on the surface of the prism contained in casing B. Filter oils if they are turbid.

<sup>a</sup> Wiley, Principles and Practice of Agricultural Analysis, 3: 339.

Press B against A and bring F back into its original position by turning it in the opposite direction. Adjust the mirror until it gives the sharpest reading. If the reading be not distinct after running water of a constant temperature through the instrument for some time, the fat is not evenly distributed on the surfaces of the prism and the process must be repeated. The instrument should be carefully adjusted by means of the standard fluid which is supplied. As the index of refraction is greatly affected by temperature, care must be used to keep the latter constant.

Use the following table to convert the degrees of the instrument into refractive indexes:

*Butyro-refractometer and indexes of refraction.\**

Reading.	Index of refraction.	Reading.	Index of refraction.	Reading.	Index of refraction.	Reading.	Index of refraction.
40.0	1.4524	50.0	1.4593	60.0	1.4659	70.0	1.4723
40.5	1.4527	50.5	1.4596	60.5	1.4662	70.5	1.4726
41.0	1.4531	51.0	1.4600	61.0	1.4665	71.0	1.4729
41.5	1.4534	51.5	1.4603	61.5	1.4668	71.5	1.4732
42.0	1.4538	52.0	1.4607	62.0	1.4672	72.0	1.4735
42.5	1.4541	52.5	1.4610	62.5	1.4675	72.5	1.4738
43.0	1.4545	53.0	1.4613	63.0	1.4678	73.0	1.4741
43.5	1.4548	53.5	1.4616	63.5	1.4681	73.5	1.4744
44.0	1.4552	54.0	1.4619	64.0	1.4685	74.0	1.4747
44.5	1.4555	54.5	1.4623	64.5	1.4688	74.5	1.4750
45.0	1.4558	55.0	1.4626	65.0	1.4691	75.0	1.4753
45.5	1.4562	55.5	1.4629	65.5	1.4694	75.5	1.4756
46.0	1.4565	56.0	1.4633	66.0	1.4697	76.0	1.4759
46.5	1.4569	56.5	1.4636	66.5	1.4700	76.5	1.4762
47.0	1.4572	57.0	1.4639	67.0	1.4704	77.0	1.4765
47.5	1.4576	57.5	1.4642	67.5	1.4707	77.5	1.4768
48.0	1.4579	58.0	1.4646	68.0	1.4710	78.0	1.4771
48.5	1.4583	58.5	1.4649	68.5	1.4713	78.5	1.4774
49.0	1.4586	59.0	1.4652	69.0	1.4717	79.0	1.4777
49.5	1.4590	59.5	1.4656	69.5	1.4720	79.5	1.4780

\* Winton, Conn. Agr. Exper. Stat. Rept., 1900, Part 2, p. 143.

#### 4. Melting Points of Fats and Fatty Acids.

##### (a) MELTING POINTS OF FATS (WILEY'S METHOD).—OFFICIAL.

###### (1) PREPARATION OF REAGENTS.

Place a piece of ice in recently boiled distilled water. Prepare a mixture of alcohol and water of the same specific gravity as the fat to be examined. This is done by boiling distilled water and 95 per cent alcohol for ten minutes to remove the gases which they may hold in solution. While still hot pour the water into the test tube until it is almost half full. Nearly fill the test tube with the hot alcohol, which is carefully poured down the side of the inclined tube to avoid too much mixing. If the alcohol be not added until the water has cooled, the mixture will contain so many air bubbles as to be unfit for use. These bubbles gather on the disk of fat as the temperature rises and finally force it to the top.

###### (2) APPARATUS.

The apparatus for determining the melting point consists of an accurate thermometer reading easily tenths of a degree; an ordinary thermometer; a tall beaker, 35 cm high and 10 cm in diameter; a test tube, 30 cm long and 3.5 cm in diameter; a stand for supporting the apparatus; some method of stirring the water in the beaker (for example, a blowing bulb of rubber and a bent glass tube reaching nearly to the bottom of the beaker). See fig. 6.

## (3) DETERMINATION.

Prepare the disks of fat as follows: Allow the melted and filtered fat to fall from a dropping tube from a height of from 15 to 20 cm onto a smooth piece of ice in boiled distilled water. The disks thus formed are from 1 to 1.5 cm in diameter and weigh about 200 mg. By pressing the ice under the water the disks are made to float on the surface, whence they are easily removed with a steel spatula, which should be cooled in the ice water before using.

The disks must be allowed to stand for two or three hours in order to obtain the normal melting point.

Place the test tube containing the alcohol and water in a tall beaker containing ice and water and leave it there until cold. Then drop the disk of fat into the tube from the spatula, and it will at once sink until it reaches a position where the density of the alcohol-water is exactly equivalent to its own. Lower a delicate thermometer into the test tube until the bulb is just above the disk. In order to secure an even temperature in all parts of the alcohol mixture in the vicinity of the disk, move the thermometer from time to time in a circle. The disk having been placed in position, slowly heat the water in the beaker, stirring it constantly by means of the blowing apparatus already described.

When the temperature of the alcohol-water mixture rises to about  $6^{\circ}\text{C}$  below the melting point the disk of fat begins to shrivel and gradually rolls up into an irregular mass. Lower the thermometer until the fat particle is even with the center of the bulb, which should be small so as to indicate only the temperature of the mixture near the fat. Give a gentle rotary motion to the thermometer bulb, and so regulate the temperature that the last  $2^{\circ}\text{C}$  of the increment require about ten minutes. The mass of the fat gradually assumes the form of a sphere, and when it has gathered in a ball make the read-

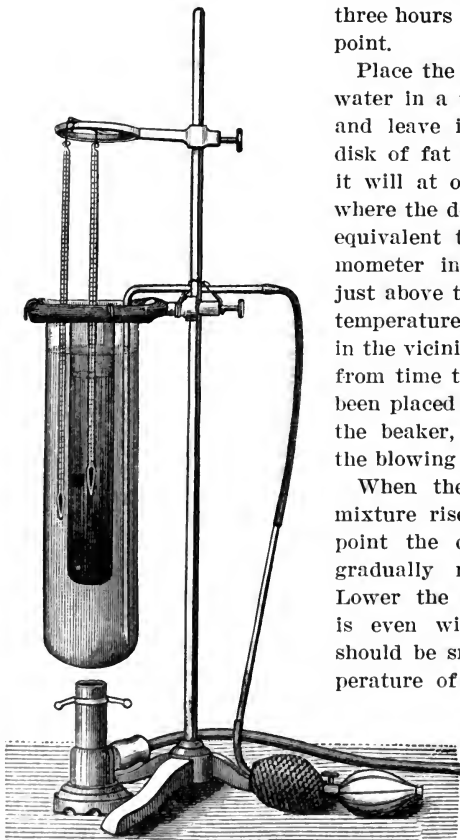


FIG. 6.—Apparatus for the determination of melting point.

ing of the thermometer. As soon as the temperature is taken remove the tube from the bath and place it again in the cooler. At once place in the bath a second tube containing alcohol and water. The test tube is of sufficiently low temperature to cool the bath to the desired point, ice water having been used as a cooler. After the first determination, which should only be a trial, the temperature of the bath should be so regulated as to reach a maximum of about  $1.5^{\circ}$  above the melting point of the fat under examination.

Do not allow the edge of the disk to touch the sides of the tube. This accident rarely happens, but in case it should take place and the disk adhere to

the sides of the tube make a new trial. Make triplicate determinations; the second and third results should agree closely. Prepare these disks a day, or at least a few hours, before using.

(b) MELTING POINT OF FATTY ACIDS.<sup>a</sup>—PROVISIONAL.

Draw the melted fatty acid into a very thin-walled capillary tube 1 or 2 inches long, according to the length of bulb of the thermometer used. Seal one end of the tube and allow the fatty acid to cool on ice for from twelve to fifteen hours. Attach to the bulb of a delicate thermometer graduated to one-fifth degree, immerse in a beaker of water, and heat very slowly. The point at which the fatty acid becomes transparent is called the melting point.

5. Titer Test.—Provisional.

(a) STANDARD THERMOMETER.

The thermometer must be graduated in tenth degrees from 10° to 60°, with a zero mark, and have an auxiliary reservoir at the upper end, also one between the zero mark and the 10° mark. The cavity in the capillary tube between the zero mark and the 10° mark must be at least 1 cm below the 10° mark, the 10° mark to be about 3 or 4 cm above the bulb, the length of the thermometer being about 15 inches over all. The thermometer is annealed for 75 hours at 450° C., and the bulb is of Jena normal 16<sup>III</sup> glass, moderately thin, so that the thermometer will be quick acting. The bulb is about 3 cm long and 6 mm in diameter. The stem of the thermometer is 6 mm in diameter and made of the best thermometer tubing, with scale etched on the stem, the graduation to be clear cut and distinct, but quite fine.

(b) DETERMINATION.

Saponify 75 grams of fat in a metal dish with 60 cc of 30 per cent sodium hydroxid (36° Baumé) and 75 cc of 95 per cent by volume alcohol or 120 cc of water. Boil to dryness, with constant stirring to prevent scorching, over a very low flame or over an iron or asbestos plate. Dissolve the dry soap in a liter of boiling water, and if alcohol has been used boil for forty minutes in order to remove it, adding sufficient water to replace that lost in boiling. Add 100 cc of 30 per cent sulphuric acid (25° Baumé) to free the fatty acids, and boil until they form a clear, transparent layer. Wash with boiling water until free from sulphuric acid, collect in a small beaker, and place on the steam bath until the water has settled and the fatty acids are clear; then decant them into a dry beaker, filter, using hot-water funnel, and dry twenty minutes at 100° C. When dried, cool the fatty acids to 15° or 20° C. above the expected titer and transfer to the titer tube, which is 25 mm in diameter and 100 mm in length (1 by 4 inches) and made of glass about 1 mm in thickness. Place in a 16-ounce saltmouth bottle of clear glass, about 70 mm in diameter and 150 mm high (2.8 by 6 inches), fitted with a cork, which is perforated so as to hold the tube rigidly when in position. Suspend the ther-

<sup>a</sup> U. S. Dept. Agr., Division of Chemistry, Bul. 13, Part 4, p. 448; Benedikt and Lewkowsitch, *Oils, Fats, and Waxes*, p. 97; Wiley, *Principles and Practice of Agricultural Analysis*, 3: 321.

mometer, graduated to  $0.10^{\circ}$  C., so that it can be used as a stirrer, and stir the mass slowly until the mercury remains stationary for thirty seconds. Then allow the thermometer to hang quietly, with the bulb in the center of the mass, and observe the rise of the mercury. The highest point to which it rises is recorded as the titer of the fatty acids.

Test the fatty acids for complete saponification as follows:

Place 3 cc in a test tube and add 15 cc of alcohol (95 per cent by volume). Bring the mixture to a boil and add an equal volume of ammonium hydroxid (0.96 sp. gr.). A clear solution should result, turbidity indicating unsaponified fat. The titer must be made at about  $20^{\circ}$  C. for all fats having a titer above  $30^{\circ}$  C. and at  $10^{\circ}$  C. below the titer for all other fats.

## 6. Iodin Absorption Number.—Official.

### (a) PREPARATION OF REAGENTS.

(1) *Hübl's iodine solution*.—Dissolve 26 grams of pure iodine in 500 cc of 95 per cent alcohol. Dissolve 30 grams of mercuric chlorid in 500 cc of 95 per cent alcohol. Filter the latter solution, if necessary, and mix the two solutions. Let the mixed solution stand twelve hours before using.

(2) *Hanus iodine solution*.—Dissolve 13.2 grams of iodine in 1,000 cc of glacial acetic acid (99.5 per cent) showing no reduction with bichromate and sulphuric acid; add enough bromine to double the halogen content determined by titration—3 cc of bromine is about the proper amount. The iodine may be dissolved by the aid of heat, but the solution should be cold when bromine is added.

(3) *Decinormal sodium thiosulphate solution*.—Dissolve 24.8 grams of chemically pure sodium thiosulphate, freshly pulverized as finely as possible and dried between filter or blotting paper, and dilute with water to 1 liter at the temperature at which the titrations are to be made.

(4) *Starch paste*.—Boil 1 gram of starch in 200 cc of distilled water for ten minutes and cool to room temperature.

(5) *Solution of potassium iodid*.—Dissolve 150 grams of potassium iodid in water and make up 1 liter.

(6) *Decinormal potassium bichromate*.—Dissolve 4.9083 grams of chemically pure potassium bichromate in distilled water and make the volume up to 1 liter at the temperature at which the titrations are to be made. The bichromate solution should be checked against pure iron.

### (b) DETERMINATION.

(1) *Standardizing the sodium thiosulphate solution*.—Place 20 cc of the potassium bichromate solution, to which has been added 10 cc of the solution of potassium iodid, in a glass-stoppered flask. Add to this 5 cc of strong hydrochloric acid. Allow the solution of sodium thiosulphate to flow slowly into the flask until the yellow color of the liquid has almost disappeared. Add a few drops of the starch paste, and with constant shaking continue to add the sodium thiosulphate solution until the blue color just disappears.

(2) *Weighing the sample*.—Weigh about one-half gram of fat or 0.250 gram of oil<sup>a</sup> on a small watch crystal or in some other suitable way. Melt the fat,

<sup>a</sup> Use from 0.100 to 0.200 gram in the case of drying oils which have a very high absorbent power.



mix thoroughly, pour onto the crystal, and allow to cool. Introduce the watch crystal into a wide-mouth 16-ounce bottle with ground-glass stopper.

(3) *Absorption of iodine in Hübl's method.*—Dissolve the fat or oil in the bottle in 10 cc of chloroform. After complete solution has taken place add 30 cc of the iodine solution in the case of fats, or from 40 to 50 cc<sup>a</sup> in the case of oils. Place the bottle in a dark place and allow to stand, with occasional shaking, for three hours.<sup>b</sup> This time must be closely adhered to in order to get good results. The excess of iodine should be at least as much as is absorbed.

(4) *Absorption of iodine in Hanus method.*—Add 25 cc of the iodine solution to the chloroform solution of the fat. Allow to stand, with occasional shaking, for thirty minutes. The excess of iodine should be at least 60 per cent of the amount added.

(5) *Titration of the unabsorbed iodine.*—Add 10 cc of the potassium iodide solution in the Hanus method, or 20 cc in the Hübl method and shake thoroughly, then add 100 cc of distilled water to the contents of the bottle, washing down any free iodine that may be noted on the stopper. Titrate the iodine with the sodium thiosulphate solution, which is added gradually, with constant shaking, until the yellow color of the solution has almost disappeared. Add a few drops of starch paste and continue the titration until the blue color has entirely disappeared. Toward the end of the reaction stopper the bottle and shake violently, so that any iodine remaining in solution in the chloroform may be taken up by the potassium iodide solution.

(6) *Standardizing the iodine solution by thiosulphate solution.*—At the time of adding the iodine solution to the fat employ two bottles of the same size as those used for the determination for conducting the operation described under paragraphs (3), (4), and (5), but without the presence of any fat. In every other respect the performance of the blank experiments should be just as described. These blank experiments must be made each time the iodine solution is used. Great care must be taken that the temperature of the solution does not change during the time of the operation, as acetic acid and alcohol have very high coefficients of expansion, and a slight change of temperature makes an appreciable difference in the strength of the solution.

Per cent of iodine absorbed:

Weight of fat taken.....	gram--	0.250
Quantity of iodine solution used.....	cc--	40.0
Thiosulphate equivalent to iodine used.....	cc--	65.0
Thiosulphate equivalent to remaining iodine.....	cc--	40.0
Thiosulphate equivalent to iodine absorbed.....	cc--	25.0

Per cent of iodine absorbed  $(25.0 \times 0.012692 \times 100)$  divided by 0.250\_\_\_ 126.92

## 7. Saponification Number or Koettstorfer Number.—Official.

### (a) PREPARATION OF REAGENTS.

(1) *Standard sodium hydroxide solution.*—Use a tenth-normal solution of sodium hydroxide. Each cubic centimeter contains 0.0040 gram of sodium hydroxide and neutralizes 0.0088 gram of butyric acid.

<sup>a</sup> F. Ulzer (J. Soc. Chem. Ind., 1898, 17: 276) says iodine should be in excess, about twice the amount that is absorbed. The solution loses strength with age, but can be used as long as 35 cc of tenth-normal thiosulphate neutralize 25 cc of iodine solution.

<sup>b</sup> The time allowed does not give the complete iodine absorption power of an oil or fat and can not be compared with determinations in which six to twelve hours have been used. It gives very satisfactory comparative results, but the time factor must be very closely observed.

(2) *Alcoholic potash solution*.—Dissolve 40 grams of chemically pure potassium hydroxid in 1 liter of 95 per cent redistilled alcohol.<sup>a</sup> The solution must be clear and the potassium hydroxid free from carbonates.

(3) *Standard acid solution*.—Prepare accurately a half-normal solution of hydrochloric acid.

(4) *Indicator*.—Dissolve 1 gram of phenolphthalein in 100 cc of 95 per cent alcohol.

#### (b) DETERMINATION.

Conduct the saponification in a wide-mouth Erlenmeyer flask holding from 250 to 300 cc. Clean thoroughly by washing with water, alcohol, and ether, wipe perfectly dry on the outside and heat for one hour at the temperature of boiling water; allow to cool and weigh.

Run in about 5 grams of the filtered melted fat by means of a pipette, and after cooling again weigh the flask and contents. Pipette 50 cc of the alcoholic potash solution into a flask by allowing it to drain for a definite time. Connect the flask with a reflux condenser and boil for 30 minutes or until the fat is completely saponified. Cool and titrate with half-normal hydrochloric acid using phenolphthalein as indicator. The Koettstorfer number (milligrams of potassium hydroxid required to saponify 1 gram of fat) is obtained as follows: Subtract the number of cubic centimeters of hydrochloric acid used to neutralize the excess of alkali after saponification from the number of cubic centimeters necessary to neutralize the 50 cc of alkali added; multiply the result by 28.06 (the number of milligrams of potassium hydroxid per cubic centimeter) and divide by the number of grams of fat used. Conduct two or three blank experiments, using the same pipette and draining for the same length of time.

#### 8. Soluble Acids.—Official.

Place the flask used in the preceding determination on a water bath and evaporate the alcohol. Add such an amount of half-normal hydrochloric acid that its volume plus the amount used in titrating for the saponification number will be 1 cc in excess of the amount required to neutralize the 50 cc of alcoholic potash added. Connect the flask with a condensing tube, 3 feet long and made of small glass tubing, and place it on the steam bath until the separated fatty acids form a clear stratum on the upper surface of the liquid. Fill to the neck with hot water and cool in ice water until the cake of fatty acids is thoroughly hardened. Pour the liquid contents of the flask through a dry weighed filter into a liter flask, taking care not to break the cake. Fill the flask again with hot water, set on steam bath until the fatty acids collect at the surface, cool by immersing in ice water, and filter the liquid again into the liter flask. Repeat this treatment with hot water three times, cooling and filtering the washings into the liter flask after each treatment. Titrate with tenth-normal alkali, using phenolphthalein as indicator.

Diminish the number of cubic centimeters of tenth-normal alkali used in this titration by 5 (corresponding to the excess of 1 cc of half-normal acid) and multiply by 0.0088 to obtain the weight of soluble acids as butyric in the amount of fat saponified; divide this by the amount of fat originally employed to obtain the percentage of soluble acids.

<sup>a</sup> The alcohol should be redistilled from potassium hydroxid on which it has been standing for some time, or with which it has been boiled for some time, using a reflux condenser.

**9. Insoluble Acids or Hehner Number.—Official.**

Allow the flask containing the cake of insoluble fatty acids from the preceding determination, and the paper through which the soluble fatty acids have been filtered, to drain and dry for twelve hours. Transfer the cake, together with as much of the fatty acids as can be removed from the filter paper, to a weighed glass evaporating dish. Then place the funnel, with the filter, in an Erlenmeyer flask and thoroughly wash the paper with absolute alcohol. Rinse the flask with the washings from the filter paper, then with pure alcohol, and transfer the filtrate and washings to the evaporating dish. Keep the dish on the steam bath until the alcohol is evaporated, dry for two hours at 100° C., cool in a desiccator, and weigh. Again place in the air bath for two hours, cool as before, and weigh. If there be any considerable decrease in weight, reheat for two hours and weigh again. The result obtained is the weight of insoluble fatty acids, from which the percentage is easily calculated.

**10. Reichert-Meissl Number or Volatile Acids.—Official.****(a) REICHERT-MEISSEL METHOD.****(1) PREPARATION OF REAGENTS.**

(a) *Sodium hydroxid solution*.—Dissolve 100 grams of sodium hydroxid, as free as possible from carbonates, in 100 cc of water, and preserve out of contact with the air. Allow to settle and use only the clear liquid.

(b) *Potassium hydroxid*.—Dissolve 100 grams of the purest potassium hydroxid in 58 cc of hot distilled water. Cool in a stoppered vessel, decant the clear solution, and preserve out of contact with the air.

(c) *Ninety-five per cent alcohol*.—Distil over sodium hydroxid.

(d) *Sulphuric acid*.—Dilute 200 cc of the strongest acid to 1,000 cc with water.

(e) *Barium hydroxid solution*.—Standardize an approximately tenth-normal solution.

(f) *Indicator*.—Dissolve 1 gram of phenolphthalein in 100 cc of 95 per cent alcohol.

(g) *Pumice stone*.—Heat small pieces to a white heat, plunge in water, and keep under water until used.

**(2) DETERMINATION.**

Melt the butter or fat and keep at about 60° C. for two or three hours, or until the water and curd have entirely separated. Pour the clear supernatant fat through a dry filter, using a hot-water funnel; if the filtered fat is not clear, filter again.

Wash the saponification flasks thoroughly with water, alcohol, and ether, wipe dry on the outside, and dry for one hour at the temperature of boiling water; cool, and weigh.

Measure 5.75 cc, equivalent to about 5 grams, of the thoroughly mixed fat into the flask with a pipette that has been warmed to about 50° C., care being taken to wipe off the adhering fat and to prevent any fat getting on the sides of the flask. Allow to stand for fifteen to twenty minutes and weigh.

Three methods of saponification are allowable:

**(a) Under pressure with alcohol.**

Place 10 cc of the 95 per cent alcohol in the flask containing the fat, which must be made of strong, well-annealed glass, capable of resisting the tension of

alcoholic vapor at  $100^{\circ}$  C., and add 2 cc of the sodium hydroxid solution. Insert a soft cork stopper in the flask, tie down with a piece of twine, and place on a water or steam bath for at least one hour (fig. 7). Gently rotate the flask from time to time, being careful that the fat does not rise on the sides of the flask to a point where it can not be reached by the alkali. Cool to room temperature before opening.

*(b) Under pressure without the use of alcohol.*

Place 2 cc of the potassium hydroxid in the flask containing the fat, which must be round bottomed and made of well-annealed glass to resist the pressure, cork, and heat as in the previous method. Rotate the flask very gently during the saponification, taking great care that none of the fat rises on the sides of the flask out of reach of the alkali. Potash makes a softer soap than soda and thus allows a complete saponification without the use of alcohol. This method avoids the danger of formation of esters and the trouble of removing the alcohol after saponification.

*(c) With a reflux condenser and the use of alcohol.*

Place 10 cc of the 95 per cent alcohol in the flask containing the fat, add 2 cc of the sodium hydroxid solution with a reflux condenser (a glass tube not less than 1 meter in length is allowable), and heat on the steam bath until the saponification is complete.

After the saponification, in case alcohol was used, remove this by dipping the flasks in a steam bath up to their necks. When the alcohol is nearly gone frothing may occur. Avoid any loss from this cause or from creeping of the soap

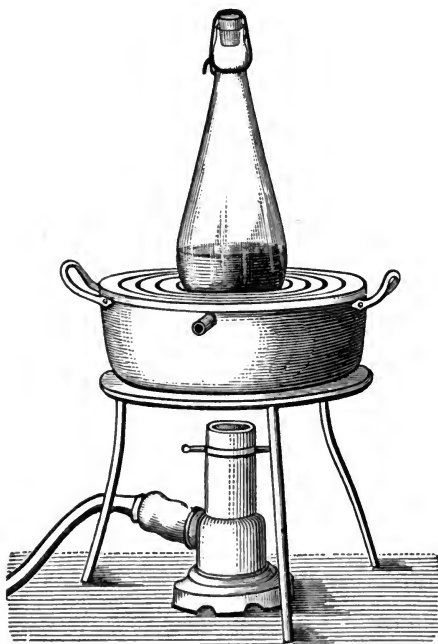


FIG. 7.—Saponification flask.

up the sides of the flask by removing from the bath and shaking to and fro. Remove the last traces of alcohol by waving the flask briskly, mouth down, to and fro, or, better, by a current of carbon dioxide free air.

Dissolve the soap by adding 135 cc of recently boiled water (or 132 cc if potassium hydroxid was used in the saponification) and warm on the water bath, with occasional shakings, until the solution is clear. Cool to from  $60^{\circ}$  to  $70^{\circ}$  C., throw in a few pieces of pumice stone, add 5 cc of the dilute sulphuric acid (or 8 cc if potassium hydroxid was used in the saponification), stopper as in the method of saponification, and heat on the water bath until the fatty acids form a clear, transparent layer on top of the water. This may take several hours. Cool to room temperature, add a few pieces of pumice stone, and connect with a glass condenser as in fig. 8.

Heat slowly with a naked flame until ebullition begins and distil, regulating

the flame in such a way as to collect 110 cc of distillate in as nearly thirty minutes as possible.

Mix this distillate, filter through a dry filter, and titrate 100 cc with the standard barium-hydroxid solution, using 0.5 cc of phenolphthalein as indicator. The red color should remain unchanged for two to three minutes.

Increase the number of cubic centimeters of tenth-normal alkali used by one-tenth, divide by the weight of fat taken, and multiply by 5 to obtain the Reichert-Meissl number. Correct result by the figure obtained in a blank experiment.

(b) LEFFMAN AND BEAM METHOD.

(1) PREPARATION OF REAGENTS.

(a) *Acid, standard barium hydroxid, indicator, and pumice stone.*—Prepare as in the previous method.

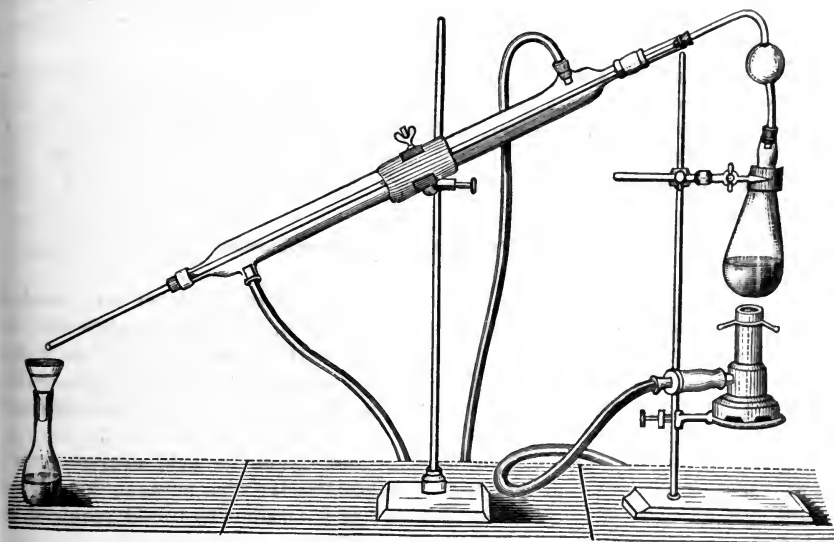


FIG. 8.—Apparatus for the distillation of volatile acids.

(b) *Glycerol-soda solution.*—Add 20 cc of the sodium-hydroxid solution used in the previous method to 180 cc of pure concentrated glycerol.

(2) DETERMINATION.

Add 20 cc of the glycerol-soda to 5 grams of the fat in a flask, weighed as in the previous method, and heat over a naked flame or hot asbestos plate until complete saponification takes place, as is shown by the mixture becoming perfectly clear. If foaming occur, shake the flask gently.

Add 135 cc of recently boiled water, drop by drop, at first, to prevent foaming, and 5 cc of the dilute sulphuric-acid solution, distil without previous melting of the fatty acids, and titrate the volatile acids as in the previous method, correcting results by the figure obtained in a blank experiment.

### 11. Liquid and Solid Fatty Acids (Muter's Method <sup>a</sup> Modified by Lane <sup>b</sup>).—Provisional.

Weigh 5 grams of oil or fat into an Erlenmeyer flask, saponify, precipitate with lead acetate, and extract with ether, as directed under determination of arachidic acid (p. 145, d). Filter the ether solution of soluble lead soap into a Muter tube or separatory funnel and decompose the soap by shaking with a 40 cc of a 1:5 solution of hydrochloric acid. The soap is completely decomposed when the ether becomes clear and colorless.

Draw off the lead chlorid from the ether solution and wash the ether free from acid. Evaporate an aliquot of this solution until it is free from ether in an atmosphere of carbon dioxid, in order to prevent the oxidation of the oleic acid, and weigh to determine the per cent of liquid acids; weigh 0.2 to 0.3 gram of this and determine the iodine number in the usual way (see page 136).

As it is very difficult to dry the oleic acid without very serious oxidation, it is just as satisfactory to determine the weight of insoluble acids by the following method:

Wash the insoluble soap left on the filter into a flask, decompose with hydrochloric acid, and heat until the fatty acids are melted. Fill the flask with hot water, cool, pour off the water, and wash again the solidified fatty acids. Dissolve in hot 95 per cent alcohol, transfer to weighed dish, remove the alcohol by evaporation, dry, weigh, and calculate the percentage of solid fatty acids.

### 12. Free Fatty Acids.<sup>c</sup>—Provisional.

Weigh 20 grams of fat or oil into a flask, add 50 cc of 95 per cent alcohol which has been neutralized with weak caustic soda, using phenolphthalein as indicator, and heat to the boiling point. Agitate the flask thoroughly in order to dissolve the free fatty acids as completely as possible. Titrate with tenth-normal alkali, agitating thoroughly until the pink color persists after vigorous shaking.

Express results either as percentage of oleic acid, as acid degree (cubic centimeters of normal alkali required to neutralize the free acids in 100 grams of oil or fat), or as acid value (milligrams of potassium hydroxid required to saturate the free acids in 1 gram of fat or oil).

1 cc of tenth-normal alkali = 0.0282 gram of oleic acid.

### 13. Acetyl Value <sup>d</sup>—Benedikt-Lewkowitsch Method.—Provisional.

Boil the oil or fat with an equal volume of acetic anhydrid for two hours, pour the mixture into a large beaker containing 500 cc of water, and boil for half an hour. To prevent bumping, pass a slow current of carbonic acid into the liquid through a finely drawn out tube reaching nearly to the bottom. Allow the mixture to separate into two layers, siphon off the water, and boil the oily layer with fresh water until it is no longer acid to litmus paper.

Separate the acetylated fat from the water and dry and filter in a drying oven.

<sup>a</sup> Muter and De Koningh, *Analyst*, 1889, 14: 61.

<sup>b</sup> J. Amer. Chem. Soc., 1893, 15: 110.

<sup>c</sup> Allen, *Commercial Organic Analysis*, 3d ed., 2: 105.

<sup>d</sup> Lewkowitsch, *J. Soc. Chem. Ind.*, 1897, 16: 503; Benedikt, *Analyse der Fette und Wachstern*, 3d ed., p. 146; Allen, *Commercial Organic Analysis*, 3d ed., 2 (1): 66.

Weigh from 2 to 4 grams of the acetylated fats into a flask and saponify with alcoholic potash as in the determination of saponification equivalent. If the distillation process is to be adopted it is not necessary to work with a standardized alcoholic potash solution, but in the filtration method, which is much shorter, the alcoholic potash must be ~~measured~~ <sup>weighed</sup> exactly. In either case evaporate the alcohol after saponification and dissolve the soap in water. Then either distil or filter as follows:

(a) DISTILLATION.

Acidify with dilute sulphuric acid (1 to 10) and distil as in the Reichert-Meissl test (page 139). As several hundred cubic centimeters must be distilled, either run a current of steam through or add portions of water from time to time. From 500 to 700 cc of distillate will be sufficient. Filter the distillates to remove any insoluble acids carried over by the steam, and titrate with tenth-normal potassium hydroxid, using phenolphthalein as indicator. Multiply the number of cubic centimeters of alkali employed by 5.61 and divide by the weight of substance used to obtain the acetyl value.

(b) FILTRATION.

Add to the soap solution a quantity of standard sulphuric acid exactly corresponding to the amount of alcoholic potash added, warm gently, filter off the free fatty acids which collect on top, wash with boiling water until the washings are no longer acid, and titrate the filtrate with tenth-normal potassium hydroxid, using phenolphthalein as indicator. Calculate the acetyl value as directed under (a).

14. Maumené Number.<sup>a</sup>—Provisional.

Place a beaker, 5 by 1.5 inches (12.7 by 3.8 cm) inside of another 6 by 3 inches (15.2 by 7.6 cm) and pack a wet mixture of asbestos and plaster of Paris tightly around the inner beaker. This, when dried, makes a hard, solid packing which radiates heat very slowly.

Remove the inner beaker, weigh into it 50 grams of fat, and note the temperature carefully. Then from a pipette which will deliver it in approximately one minute add 10 cc of the strongest sulphuric acid which is at the same temperature as the oil. Stir the oil and acid with an accurate thermometer while the acid is being introduced, then hold the thermometer bulb carefully in the center of the mixture, and when the mercury reaches the highest point note the reading. It is easy to determine this point, as the column of mercury remains stationary for some time. Do not read the temperature too soon, as some oils take considerable time to reach their maximum point.

The difference between the initial and the final reading expressed in degrees Centigrade is the Maumené number.

Great care must be taken to use acid of maximum strength. With the semi-drying oils, such as cottonseed, the use of this strength of acid will cause foaming and make it almost impossible to obtain the correct rise of temperature. With such oils either a weaker acid must be used and the results compared with the rise of temperature with water or a dilution with paraffin oil is made. It is always best to test the apparatus and acid by use of water and oils of known purity. In reporting results, the rise of temperature with water should be stated, otherwise they possess no comparative value.

<sup>a</sup> Allen, Commercial Organic Analysis, 3d ed., 2 (1): 76.

15. Phytosterol and Cholesterol.<sup>a</sup>—Provisional.

Boil 50 grams of fat or oil in a flask having a reflux condenser with 75 cc of 95 per cent alcohol for five minutes and separate the alcoholic solution. Repeat with another portion of alcohol and separate. Mix the alcoholic solution with 15 cc of 30 per cent sodium hydroxid and boil in a flask with a condensation tube until one-fourth of the alcohol is evaporated. Evaporate nearly to dryness in a porcelain dish and shake the residue with ether. Evaporate the ethereal solution to dryness, take up with a little ether, filter, again evaporate, dissolve in hot 95 per cent alcohol, and allow to crystallize.

Cholesterol can easily be distinguished from phytosterol by the form and grouping of the crystals, also by the melting point, which is 146° C.,<sup>b</sup> while that of phytosterol is from 130° to 137.5° C.<sup>c</sup> The crystals of phytosterol separated from hot alcohol appear as tufts of needles. Cholesterol is characteristic of animal fats; it crystallizes in thin rhombic tables.

16. Unsaponifiable Residue.<sup>d</sup>—Provisional.

Saponify 5 grams of oil or fat with alcoholic potassium hydroxid and remove the alcohol by evaporation. Wash into separatory funnel with from 70 to 100 cc of water and extract with from 50 to 60 cc of ether. In case the two liquids do not separate, a few cubic centimeters of alcohol may be added. Separate the water solution and wash the ether with water containing a few drops of sodium hydroxid. Again extract the soap solution and washings with ether and evaporate the combined extracts to dryness. In most cases it is advisable to add a little alcoholic potassium hydroxid to the residue and heat in order to saponify any traces of fats left unsaponified and extract again with ether. Transfer to a weighed dish and dry as quickly as possible in a water oven.

Many of the hydrocarbon oils are volatile at 100° C., so that the drying should not be carried any further than necessary. With resin oil, paraffin wax, and the denser mineral oils there is little danger of loss at 100° C.

On account of the solubility of soap in ether and petroleum ether it is well to wash the residue with warm water containing a little phenolphthalein. If the reaction is alkaline, soap is present and the residue must be further purified.

## 17. Qualitative Tests.

## (a) RESIN OIL.—PROVISIONAL

Polarize the pure oil or a definite dilution with petroleum ether in a 200 mm tube. Resin oil has a polarization in a 200 mm tube of from +30° to +40° on the sugar scale (Schmidt and Haensch) while other oils read between +1° and -1°.

(b) HALPHEN <sup>e</sup> REACTION FOR COTTON-SEED OIL.—PROVISIONAL

Mix carbon disulphid, containing about 1 per cent of sulphur in solution with an equal volume of amyl alcohol. Mix equal volumes of this reagent and

<sup>a</sup> Forster and Reichelmann, *Analyst*, 1897, 22: 131; E. Salkowski, *Zts. anal. Chem.*, 1887, 26: 557; E. Von Raumer, *Zts. angew. Chem.*, 1898, 13: 555-556; J. Soc. Chem. Ind., 1898, 17: 774; H. Kreis and O. Wolf, *J. Soc. Chem. Ind.*, 1898, 17: 1075.

<sup>b</sup> E. Salkowski, *Zts. anal. Chem.*, 1887, 26: 557.

<sup>c</sup> Bömer, *Zts. Nahr. Genussm.*, 1898, 1: 81.

<sup>d</sup> Allen, *Commercial Organic Analysis*, 3d ed., 2 (1): 112.

<sup>e</sup> J. Pharm. Chim., 1897, 6: 390; *Analyst*, 1897, 22: 326; Allen, *Commercial Organic Analysis*, 3d ed., 2 (1): 143; Winton, *Conn. Agr. Exper. Stat. Rept.*, 1900, Part 2, p. 144.



the oil under examination, and heat in a bath of boiling saturated brine for from 1 to 2 hours. In the presence of as little as 1 per cent of cotton-seed oil, a characteristic red or orange-red color is produced.

Lard and lard oil from animals fed on cotton-seed meal will give a faint reaction; the fatty acids also give this reaction.

The depth of color is proportional, to a certain extent, to the amount of oil present, and by making comparative tests with cotton-seed oil some idea as to the amount present can be obtained, but it must be remembered that different oils react with different intensities, and oils which have been heated from 200° to 210° C.<sup>a</sup> react with greatly diminished intensity. Heating 10 minutes at 250° C. renders cotton-seed oil incapable of giving a reaction.<sup>b</sup>

(c) BECHI OR SILVER NITRATE TEST FOR COTTON-SEED OIL.—PROVISIONAL.

(1) PREPARATION OF REAGENT.<sup>c</sup>

Dissolve 2 grams of silver nitrate in 200 cc of 95 per cent alcohol and 40 cc of ether, adding 1 drop of nitric acid.

(2) DETERMINATION.

Mix 10 cc of oil or melted fat, 5 cc of reagent, and 10 cc of amyl alcohol<sup>d</sup> in a test tube. Divide, heat one half in a boiling water bath for ten minutes and compare with portion not heated. Any blackening due to reduced silver shows presence of cotton-seed oil.

Other oils which have become rancid<sup>e</sup> and lards which have been steamed or heated at high temperature contain decomposition products which have a reducing action on silver nitrate. Some salad oils which contain no cotton-seed oil, according to the Halphen test, give a brown coloration with the Bechi reagent and in some cases reduce silver. Such oils when purified give no reaction, and therefore the oils or fats should always be purified before testing. To effect this, heat from 20 to 30 grams of the sample on a water bath for a few minutes together with 25 cc of 95 per cent alcohol, shake thoroughly, decant as much of the alcohol as possible, wash with 2 per cent nitric acid and finally with water. Heating the oils or fats to 100° C. or simply washing with 2 per cent nitric acid is not sufficient except in a few cases.

(d) RENARD'S TEST<sup>f</sup> FOR PEANUT OIL AS MODIFIED BY TOLMAN.—PROVISIONAL.

Weigh 20 grams of oil into an Erlenmeyer flask. Saponify with alcoholic potash, neutralize exactly with dilute acetic acid, using phenolphthalein as indicator, and wash into a 500 cc flask containing a boiling mixture of 100 cc of water and 120 cc of a 20 per cent lead acetate solution. Boil for a minute and then cool the precipitated soap by immersing the flask in water, occasionally giving it a whirling motion to cause the soap to stick to the sides of the flask. After the

<sup>a</sup> Allen, *Commercial Organic Analysis*, 3d ed., 2 (1): 143.

<sup>b</sup> Holde and Pelgry, *J. Soc. Chem. Ind.*, 1899, 18: 711.

<sup>c</sup> Pearmain and Moor, *Allen's Commercial Organic Analysis*, 3d ed., 2 (1): 143; Wesson, *J. Amer. Chem. Soc.*, 1895, 17: 724.

<sup>d</sup> The addition of amyl alcohol is not necessary, but is very convenient, as it dissolves the oils or fats, which then mix with the reagent much better.

<sup>e</sup> Wesson, *J. Amer. Chem. Soc.*, 1895, 17: 724; Winton, *Conn. Agr. Exper. Stat. Rept.*, 1900, Part 2, p. 143.

<sup>f</sup> *Comp. rend.*, 1871, 73: 1330; Benedikt and Lewkowitsch, *Oils, Fats, and Waxes*, p. 365.

flask has cooled, the water and excess of lead can be poured off and the soap washed with cold water and with 90 per cent (by volume) alcohol. Add 200 cc of ether, cork, and allow to stand for some time until the soap is disintegrated; heat on the water bath, using a reflux condenser, and boil for about five minutes.<sup>a</sup> In the oils most of the soap will be dissolved, while in lards, which contain much stearin, part will be left undissolved. Cool the ether solution of soap to from 15° to 17° C. and let stand until all the insoluble soaps have crystallized out (about twelve hours).

Filter and thoroughly wash the precipitate with ether. Save the filtrate for the determination of the iodine number of the liquid fatty acids by the Muter method (page 142). Wash the soaps on the filter back into the flask by means of a stream of hot water acidified with hydrochloric acid. Add an excess of dilute hydrochloric acid, partially fill the flask with hot water, and heat until fatty acids form a clear oily layer. Fill the flask with hot water, allow the fatty acids to harden and separate from the precipitated lead chlorid, wash, drain, repeat washing with hot water, and dissolve the fatty acids in 100 cc of boiling 90 per cent by volume alcohol. Cool to 15° C., shaking thoroughly to aid crystallization.

From 5 to 10 per cent of peanut oil can be detected by this method, as it effects a complete separation of the soluble acids from the insoluble, which interfere with the crystallization of the arachidic acid. Filter, wash the precipitate twice with 10 cc of 90 per cent by volume alcohol, and then with alcohol 70 per cent by volume. Dissolve off the filter with boiling absolute alcohol, evaporate to dryness in a weighed dish, dry, and weigh. Add to this weight 0.0025 gram for each 10 cc of 90 per cent alcohol used in the crystallization and washing if done at 15° C.; if done at 20° add 0.0045 gram for each 10 cc. The melting point of arachidic acid thus obtained is between 71° and 72° C. Twenty times the weight of arachidic acid will give the approximate amount of peanut oil present. No examination for adulterants in olive oil is complete without making the test for peanut oil. Arachidic acid has a characteristic structure and can be detected by the microscope.

#### (e) BAUDOUIN TEST FOR SESAME OIL.—PROVISIONAL.

Dissolve 0.1 gram of finely powdered sugar in 10 cc of hydrochloric acid (sp. gr. 1.20), add 20 cc of the oil to be tested, shake thoroughly for a minute, and allow to stand. The aqueous solution separates almost at once. In the presence of even a very small admixture of sesame oil this is colored crimson. Some olive oils give a slight pink coloration with this reagent, but they are not hard to distinguish if comparative tests with sesame oil are made.

#### (f) VILLAVECCHIA <sup>b</sup> TEST FOR SESAME OIL.—PROVISIONAL.

Add 2 grams of furfural to 100 cc of alcohol (95 per cent) and mix thoroughly 0.1 cc of this solution, 10 cc of hydrochloric acid (sp. gr. 1.20), and 10 cc of oil by shaking them together in a test tube. The same color is developed as when sugar is used, as in the Baudouin test. Villavecchia explained this reaction on the basis that furfural is formed by the action of levulose and hydrochloric acid and therefore substituted furfural for sucrose. As furfural gives a violet tint with hydrochloric acid it is necessary to use the very dilute solution specified in the method.

<sup>a</sup> Process used by N. J. Lane in his modification of Muter's method; J. Amer. Chem. Soc., 1893, 13: 110.

<sup>b</sup> Villavecchia and Fabris, J. Soc. Chem. Ind., 1893, 12: 67; 1894, 13: 69. Benedikt and Lewkowitsch, Oils, Fats, and Waxes, p. 385.

(g) BELFIELD <sup>a</sup> MICROSCOPIC TEST FOR BEEF FAT.—PROVISIONAL.

Dissolve from 2 to 5 grams of oil or fat in a test tube with about 10 cc of ether, plug the test tube lightly with cotton, and allow to stand 15 or more hours in a moderately cool place.

The most characteristic crystals are obtained when the crystallization proceeds slowly and at a temperature of 22° to 24° C. The first crop of crystals may be examined and the mother liquor separated and set aside for further crystallization.

Gladding <sup>b</sup> has suggested the following procedure to eliminate the olein:

Dissolve in an Erlenmeyer flask 5 grams of melted fat in 10 cc of absolute alcohol and 5 cc of ether, stopper with cotton, and place in ice water for about one-half hour, until the more crystallizable portions of the fat have separated. Separate the crystalline part by filtration through a filter paper moistened with alcohol and wash with the alcohol-ether mixture. After drying in the air for some time dissolve the crystals from the paper by means of ether and then treat as described in the preceding method. When the crystals are ready to be examined remove a drop with a pipette, place on a slide, add a drop of cotton or olive oil, and press a cover glass gently over it.

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<sup>a</sup> U. S. Dept. Agr., Division of Chemistry, Bul. 13, Part 4, p. 449; Gladding, J. Amer. Chem. Soc., 1896, 18:189; Wiley, Principles and Practice of Agricultural Analysis, 3: 345, 346; Winton, Conn. Agr. Exper. Stat. Rept., 1900, Part 2, p. 145.

<sup>b</sup> J. Amer. Chem. Soc., 1896, 18: 189.

## **XX. METHODS FOR THE ANALYSIS OF COCOA AND COCOA PRODUCTS.**

See Appendix, page 254, for provisional methods adopted at the meeting of 1907.

## **XXI. METHODS FOR THE ANALYSIS OF TEA.—PROVISIONAL.**

### **1. Detection of Stems, Dust, and Foreign Leaves.**

Place 1 gram of tea in a 300 cc casserole, add 200 cc of water, and boil for fifteen minutes. This treatment will cause the leaves to unroll, and a megascopic examination will reveal the presence or absence of stems or dust, while the leaves will be in condition for examination as to their form and structure.

### **2. Detection of Facing.**

Tea is faced by treating it with pigments, graphite, catechu, etc. Mineral pigments can be detected in the ash, or the tea may be shaken up with a large volume of water, and the water separated from the leaves by a sieve, when the insoluble mineral substances used in facing will settle, and can be removed by filtration for further examination, the catechu and other soluble substances being in the filtrate.

### **3. Preparation of Sample.**

Grind the material and pass it through a sieve having perforations 0.5 mm in diameter.

### **4. Moisture.**

Proceed as directed under "VI. General Methods," page 38.

### **5. Soluble Solids—Krauch Method.**

Treat 20 grams of tea with 400 cc of water and heat on a boiling-water bath for six hours. Filter through a tared filter, wash with water until the filtrate measures 1,000 cc, dry, and weigh the residue. Determine the water soluble substance by difference.

### **6. Ash.**

Proceed as directed under "VI. General Methods," page 38.

### **7. Soluble Ash.**

Proceed as directed under "X. Saccharine Products," 3 (c), page 68.

### **8. Ash Insoluble in Acid.**

Proceed as directed under "XXIV. Spices," on page 162.

### **9. Alkalinity of Ash.**

Proceed as directed under "X. Saccharine Products," 3 (d) (e), page 69.

### 10. Phosphates.

Determine  $P_2O_5$  in the soluble and insoluble ash as directed under "I. Fertilizers," page 1.

### 11. Petroleum Ether Extract.

Proceed as directed under "XXII. Coffee," on page 154, paragraph 22.

### 12. Protein.

Proceed as directed under "XXII. Coffee," on page 153, paragraph 15.

### 13. Crude Fiber.

Proceed as directed under "VI. General Methods," page 56.

### 14. Volatile Oil.

Distil 100 grams of tea with 800 cc of water, extract the filtrate several times with petroleum ether, evaporate the combined petroleum ether extracts at the room temperature, dry in a desiccator, and weigh.

### 15. Caffein (or Thein)—Dvorkovitsch Method.<sup>a</sup>

Digest 10 grams of the powdered tea with 200 cc of boiling water for five minutes and decant the solution; repeat the treatment twice and boil the residue twice with 200 cc of water. Make up the combined solutions to 1,000 cc and extract a portion with petroleum ether to remove the fat, etc. To 600 cc of the fat-free solution (equivalent to 6 grams of tea) add 100 cc of 4 per cent barium hydroxid, mix, and filter. To 583 cc of the filtrate (equivalent to 5 grams of tea) add 100 cc of a 20 per cent solution of sodium chlorid, and extract three times with chloroform. Distil the greater part of the chloroform from the combined extracts, place the residue in a tared dish, evaporate the remainder of the chloroform, dry at  $100^{\circ}C.$ , and weigh. The caffein is usually of sufficient purity to render a nitrogen determination unnecessary.

### 16. Tannin—Proctor's Modification of Löwenthal's Method.<sup>b</sup>

#### (a) PREPARATION OF REAGENTS.

(1) *Potassium permanganate*.—Make up a solution containing 1.33 grams per liter.

(2) *Tenth-normal oxalic acid*.—Make up a solution containing 6.3 grams per liter.

(3) *Indigo carmine*.—Make up a solution containing 6 grams of indigo carmen (free from indigo blue) and 50 cc of concentrated sulphuric acid per liter.

(4) *Gelatin solution*.—Prepare by soaking 25 grams of gelatin for one hour in a saturated sodium chlorid solution, heat until the gelatin is dissolved, and make up to 1 liter after cooling.

(5) *Mixture*.—Combine 975 cc of saturated sodium chlorid solution and 25 cc of concentrated sulphuric acid.

(6) *Powdered kaolin*.

<sup>a</sup> Ber. d. chem. Ges., 1891, 24: 1945.

<sup>b</sup> U. S. Dept. Agr., Division of Chemistry, Bul. 13, Part 7, p. S90.

## (b) DETERMINATION.

Obtain the value of the potassium permanganate in terms of the oxalic acid. Boil 5 grams of the tea for half an hour with 400 cc of water; cool, transfer to a graduated 500 cc flask, and make up to the mark. To 10 cc of the infusion (filtered if not clear) add 25 cc of the indigo carmine solution and about 750 cc of water. Add from a burette the potassium permanganate solution, a little at a time while stirring, until the color becomes light green, then cautiously, drop by drop, until the color changes to bright yellow or, further, to a faint pink at the rim. The number of cubic centimeters of permanganate used furnishes the value  $a$  of the formula given below.

Mix 100 cc of the clear infusion of tea with 50 cc of gelatin solution, 100 cc of salt acid solution, and 10 grams of kaolin, and shake several minutes in a corked flask. After settling decant through a filter. Mix 25 cc of the filtrate (corresponding to 10 cc of the original infusion) with 25 cc of the indigo solution and about 750 cc of water, and titrate with permanganate as before. The number of cubic centimeters of permanganate used gives the value  $b$ ;  $a-b=c$ ;  $c$  equals the amount of permanganate required to oxidize the tannin. Assume that 0.04157 gram of tannin (gallotannic acid) is equivalent to 0.063 gram of oxalic acid.

## XXII. METHODS FOR THE ANALYSIS OF COFFEE.—PROVISIONAL.

### RAW COFFEE.

The proximate examination of raw coffee is of no value in determining its quality, since no substitutes are on the market, and as it is not adulterated with other substances for the purpose of increasing its volume or weight, the proximate analysis is not made.

The inferior grades are sometimes colored to imitate the better grades. Pigments, such as ocher, litharge, lead chromate, and chromic oxid, and organic colors, such as indigo, turmeric, azo yellow, malachite green, methyl green, etc., have been used for this purpose. The mineral colors can be identified in the ash or removed from the bean by shaking with water, in which case the soluble colors can be identified in the solution and the insoluble ones in the residue.

### ROASTED COFFEE.

#### 1. Examination of the Whole Beans.

Examine megascopically in order to detect foreign substances. Artificial coffee beans are apparent from their exact regularity of form. Coffee pellets are made from roasted wheat mash and are of a brown color, possessing a very characteristic ellipsoidal form. Roasted legumes and lumps of chicory are sometimes present and are easily identified by the practiced eye. Coffee is glazed by treating the raw bean with molasses, sugar solution, glycerin, or fats and waxes, and then roasting.

#### 2. Detection of Sugar Glazing—Stutzer and Reitnair Method.<sup>a</sup>

Treat 20 grams of the whole beans with 500 cc of water in a liter flask and shake vigorously for five minutes. Bring the volume to the mark with water, mix, filter, and determine the solids and ash in 50 cc of the filtrate.

#### 3. Detection of Glycerol.

Proceed as directed under "XIII. Wine," section 3, page 83, using a portion of the filtrate from the preceding determination.

#### 4. Detection of Fats and Waxes—Späth Method.<sup>b</sup>

Treat 100 to 200 grams of the beans with low boiling petroleum ether for ten minutes, pour off the petroleum ether and repeat the process. Filter the combined petroleum ether extracts, evaporate, and determine the saponification number and index of refraction of residue, as directed under "XIX. Edible Fats and Oils," pages 131 and 137.

#### 5. Preparation of Sample.

Grind the sample and pass it through a sieve having holes 0.5 mm in diameter.

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<sup>a</sup> Pharm. Centralh., 22: 134.

<sup>b</sup> Forschungsberichte über Lebensmittel, 1895, 2: 223.



**6. Moisture.**

Proceed as directed under "VI. General Methods," on page 38.

**7. Soluble Solids—Winston's Method.**

Place 4 grams of the sample in a 200 cc flask, add water to the mark, and allow the mass to macerate for eight hours, with occasional shaking; let stand sixteen hours longer without shaking, filter, evaporate 50 cc of the filtrate to dryness in a flat-bottomed dish, dry at 100° C., and weigh.

**8. Ash.**

Proceed as directed under "VI. General Methods," on page 38.

**9. Soluble Ash.**

Proceed as directed under "X. Saccharine Products," on page 68 (c).

**10. Ash Insoluble in Acid.**

Proceed as directed under "XXIV. Spices," page 162.

**11. Chlorin.**

Proceed as directed under "III. Inorganic Plant Constituents," page 23.

**12. Alkalinity of Soluble Ash.**

Proceed as directed under "X. Saccharine Products," page 69 (d).

**13. Soluble Phosphates in the Ash.**

Determine phosphoric acid ( $P_2O_5$ ) in soluble ash, as directed under "I. Fertilizers," page 1.

**14. Insoluble Phosphates in the Ash.**

Determine phosphoric acid ( $P_2O_5$ ) in the insoluble ash as directed under "I. Fertilizers," page 1.

**15. Protein.**

Determine nitrogen in 3 grams of the sample by the Kjeldahl or Gunning method (page 5, under "I. Fertilizers"). This gives the total nitrogen due to both the proteids and the caffeine. To obtain the protein nitrogen, subtract from the total nitrogen the nitrogen due to caffeine, obtained by direct determination on the separated caffeine or by calculation (caffeine divided by 3.464 gives nitrogen). Multiply by 6.25 to obtain the amount of protein.

**16. Caffein—Hilger and Fricke Method.<sup>a</sup>**

To from 5 to 10 grams of coffee add 100 cc of water and boil, filter, and treat the residue twice more with boiling water. Add to the united filtrates an excess of lead acetate, filter, and wash. Treat the filtrate with hydrogen sulphid to

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<sup>a</sup> Arch. Pharm., 1885, p. 827.

remove the excess of lead, filter, wash, and evaporate the filtrate to dryness in a Hoffmeister Schälchen with some sand and a little magnesia. Crush the schälchen between the filter paper, place in a continuous extraction apparatus, and extract with chloroform until exhausted. Dry the chloroform extract at 100° C. and weigh as caffen. If the caffen does not appear to be pure, determine nitrogen in the residue by the Kjeldahl or Gunning method; multiply the amount of nitrogen found by 3.464 to obtain the amount of caffen present.

#### 17. Crude Fiber.

Proceed as directed under "VI. General Methods," page 56.

#### 18. Crude Starch by Direct Inversion.

Use 4 grams of the sample and proceed as directed under "VI. General Methods," page 53, section 8 (a).

#### 19. Starch.

Use 4 grams of the sample and employ the diastase method as given on page 53, under "VI. General Methods," section 8 (b).

#### 20. Sucrose.

Use half the normal weight and proceed as directed under "VI. General Methods," page 40, section 6 (c).

#### 21. Reducing Sugars.

Use 100 cc of the solution prepared as directed under "7. Soluble Solids," and proceed as directed under "VI. General Methods," page 42, section 7 (a). Calculate results as dextrose.

#### 22. Petroleum Ether Extract.

Dry 2 grams of coffee at 100° C., extract with petroleum ether (boiling point 35° to 50° C.) for 16 hours, evaporate the solvent, dry the residue at 100° C., and weigh.

#### 23. Ten Per Cent Extract—McGill Method.

Weigh into a tared flask the equivalent of 10 grams of the dried substance, add water until the contents of the flask weigh 110 grams, connect with a reflux condenser and heat, beginning the boiling in 10 to 15 minutes. Boil for 1 hour, cool for 15 minutes, weigh again, making up any loss by the addition of water, filter, and take the specific gravity of the filtrate at 15° C.

According to McGill, a 10 per cent extract of pure coffee has a specific gravity of 1.00986 at 15° C., and under the same treatment chicory gives an extract with a specific gravity of 1.02821. In mixtures of coffee and chicory the approximate percentage of chicory may be calculated by the following formula:

$$\text{Per cent of chicory} = 100 - \frac{(1.02821 - \text{sp. gr.})}{0.01835}$$

The index of refraction of the above solution may be taken with the Zeiss immersion refractometer or with the Abbe refractometer.

With a 10 per cent coffee extract,  $n_D^{20} = 1.3377$ .

With a 10 per cent chicory extract,  $n_D^{20} = 1.3448$ .

Determinations of the solids, ash, sugar, nitrogen, etc., may be made in the 10 per cent extract, if desired.

**24. Caffetannic Acid—Krug's Method.<sup>a</sup>**

Treat 2 grams of the coffee with 10 cc of water and digest for 36 hours; add 25 cc of 90 per cent alcohol and digest 24 hours more, filter, and wash with 90 per cent alcohol. The filtrate contains tannin, caffeine, color, and fat. Heat the filtrate to the boiling point and add a saturated solution of lead acetate. If this is carefully done, a caffetannate of lead will be precipitated containing 49 per cent of lead. As soon as the precipitate has become flocculent, collect on a tared filter, wash with 90 per cent alcohol until free from lead, wash with ether, dry, and weigh. The precipitate has the following composition:  $Pb_3(C_{15}H_{15}O_8)_2$ . The weight of the precipitate multiplied by 0.51597 gives the weight of the caffetannic acid.

**25. Megascopic Examination of Ground Coffee.**

Roasted cereals have a glossy appearance entirely different from that of roasted coffee, and if roasted legumes are present, fragments of the hulls may be seen. Mix a portion of the sample with cold water; fragments of pure coffee, if not overroasted, will float, while common adulterants will sink. Examine the latter microscopically.

**26. Microscopic Examination of Ground Coffee.**

Examine for cereals, chicory, etc.

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<sup>a</sup> U. S. Dept. Agr., Division of Chemistry, Bul. 13, Part 7, p. 908.

## XXIII. METHODS FOR THE ANALYSIS OF FLAVORING EXTRACTS.—PROVISIONAL.

### VANILLA AND ITS SUBSTITUTES.

#### 1. Specific Gravity.

Determine as directed under "XIII. Wine," on page 83.

#### 2. Alcohol.

Determine as directed under "XV. Distilled Liquors," on page 95.

#### 3. Glycerol.

Determine as directed under "XIII. Wine," on page 83.

#### 4. Determination and Identification of Vanillin, Coumarin, and Acetanilid.

(a) HESS AND PRESCOTT METHOD MODIFIED BY WINTON AND BAILEY.<sup>a</sup>

Weigh 25 grams into a 200 cc beaker with marks showing volumes of 25 and 50 cc. Dilute to the 50 cc mark and evaporate in a water bath to 25 cc at a temperature in the bath of not more than 70° C. Dilute a second time to 50 cc and evaporate to 25 cc. Add normal lead acetate solution drop by drop until no more precipitate forms. Stir with a glass rod to facilitate flocculation of the precipitate, filter through a moistened filter, and wash three times with hot water, taking care that the total filtrate does not measure more than 50 cc. Cool the filtrate and shake with 20 cc of ether in a separatory funnel. Remove the ether to another separatory funnel and repeat the shaking of the aqueous liquid with ether three times, using 15 cc each time. Shake the combined ether solutions four or five times with 2 per cent ammonium hydroxid, using 10 cc for the first shaking and 5 cc for each subsequent shaking. Set aside the combined ammoniacal solutions for the determination of vanillin.

Wash the ether solution into a weighed dish and allow the ether to evaporate at the room temperature. Dry in a desiccator, and weigh. Stir the residue for fifteen minutes with 15 cc of petroleum ether (boiling point 30° to 40° C.) and decant the clear liquid into a beaker. Repeat the extraction with petroleum ether two or three times. Allow the residue to stand in the air until apparently dry, completing the drying in a desiccator. Weigh, and deduct the weight from the weight of the residue obtained after the ether evaporation, thus obtaining the weight of the coumarin. The petroleum ether residue, if acetanilid, should melt at about 112° C. and respond to Ritser's tests (p. 158, d).

Allow the petroleum ether extract to evaporate at room temperature. If it is pure coumarin, it should have a melting point within a few degrees of 67° C. and respond to Leach's test. (See page 157, paragraph (c).)

<sup>a</sup> J. Amer. Chem. Soc., 1899, 21: 256; 1902, 24: 1128; 1905, 27: 719.

Slightly acidulate the ammoniacal solution reserved for vanillin with 10 per cent hydrochloric acid. Cool, and shake out in a separatory funnel with four portions of ether, as described for the first ether extraction. Evaporate the ether at room temperature in a weighed dish, dry over sulphuric acid, and weigh.

If acetanilid has not been previously detected, this residue should be pure vanillin with a melting point within a few degrees of 80° C.

If acetanilid has been detected, dissolve the residue in 15 cc of 10 per cent ammonium hydroxid and shake twice with an equal volume of ether. Evaporate the ether solution at room temperature, dry in a desiccator, and weigh. Deduct this weight from the previous weight, thus obtaining the weight of pure vanillin. The total weight of the acetanilid is obtained by adding the weight of this last extract to that of the residue previously obtained and identified as acetanilid.

In doubtful cases the ammoniacal solution should be acidified, shaken out with ether, and the melting point of the vanillin, obtained by evaporation at room temperature, determined.

(b) COLORIMETRIC METHOD FOR THE DETERMINATION OF VANILLIN.<sup>a</sup>

(1) PREPARATION OF REAGENTS.

(a) *Vanillin*.—Prepare a standard solution by dissolving 50 mg in 25 cc of alcohol and diluting to 100 cc with water.

(b) *Moist lead hydrate*.—Dissolve 200 grams of lead acetate in 850 cc of water, filter, and add an excess of potassium or sodium hydroxid. Let the precipitate settle and wash thoroughly by decantation with repeated portions of water until perfectly neutral. Keep in 500 cc of water in the reagent bottle, and shake to form an emulsion-like mixture before adding to decolorize.

(2) DETERMINATION.

Measure 2 cc of the vanilla extract into a test tube and add about 5 cc of the lead hydrate; mix thoroughly, pour upon a small wet filter, collect filtrate and washings in a 50 cc graduated Nessler tube; add an excess of bromin water (3 or 4 drops) and sufficient freshly prepared 10 per cent ferrous sulphate solution to produce the maximum bluish-green color that will result if vanillin is present, and fill to the mark with water.

Compare with solutions containing a known amount of vanillin treated as directed above.

(c) DETECTION OF COUMARIN—LEACH'S TEST.<sup>b</sup>

Dissolve a few of the crystals or the small crystalline residue in a few drops of hot water, filter if necessary, and add to the clear solution a few drops of tenth-normal iodine in potassium iodid. In the presence of coumarin a brown precipitate will form, which on stirring or shaking will soon gather in dark-green flecks, leaving a clear brown solution. The reaction is especially marked if the iodine reagent is applied with a glass rod to the few drops of solution to be tested on a white plate or tile.

<sup>a</sup> Leach, Food Inspection and Analysis, p. 735.

<sup>b</sup> Ibid., p. 737.

(d) DETECTION OF ACETANILID.<sup>a</sup>

Boil the acetanilid, obtained as described under (a), in a small beaker for two or three minutes with 2 to 3 cc of concentrated hydrochloric acid, cool, divide into three portions, and test in small tubes (4 to 5 mm inside diameter), as follows:

## RITSERT'S TESTS.

To one portion add carefully 1 to 3 drops of a solution of chlorinated lime (1:200) in such a manner that the two solutions do not mix. A beautiful blue color formed at the juncture of the two liquids indicates acetanilid.

(2) To another portion add a small drop of potassium permanganate solution. A clear green color is formed if any appreciable amount of acetanilid is present.

(3) Mix the third portion with a small drop of 3 per cent chromic acid solution. Acetanilid gives a yellow-green solution, changing to dark green on standing five minutes, and forming a dark blue precipitate on addition of a drop of caustic potash solution.

These tests are conclusive only when taken in conjunction with the melting point.

## 5. Total Solids.

Weigh about 25 grams of the extract into a large, flat-bottomed dish which contains enough freshly ignited asbestos to absorb it; dry for twenty to twenty-four hours in a water-jacketed oven.

## 6. Determination of Ash.

Evaporate 10 grams of the extract and determine the ash as directed under "VI. General Methods," page 38.

## 7. Examination of Ash.

Proceed as directed under "III. Inorganic Plant Constituents," page 21, or under "XXVI. Baking Powder, etc.," page 177.

## 8. Sucrose.

Determine as directed under "XIII. Wine," on page 87, under section 17.

## 9. Detection of Vanilla Resins.

## (a) GENERAL DISCUSSION.

The most important fragrant principle of the vanilla bean and of true vanilla extract is vanillin, or hydroxymethoxybenzoic aldehyde. It is not, however, the only fragrant or valuable constituent of the vanilla bean and true vanilla extracts. The artificial vanillin is identical with the vanillin contained in the vanilla bean, but the bean also contains, among the many extractive matters which enter into the food value and fragrance of the extract, certain resins, which can be identified with certainty by a number of reactions. If negative results are obtained by these tests, it is evident that the extract was not made from true vanilla beans.

Vanilla beans contain from 4 to 11 per cent of these resins, which vary from dark red to brown in color and furnish about one-half of the color of the

<sup>a</sup> Pharm. Ztg., 1888, 33: 383; Abs. Zts. anal. Chem., 1888, 27: 667.

extract. The resins are soluble in 50 per cent alcohol, so that in high-grade extracts, in which sufficient alcohol is used, all of the resins are kept in solution. In cheap extracts, in which as little as 20 per cent of alcohol by volume is sometimes employed, an alkali—usually potassium bicarbonate—is added to aid in dissolving the resins, gums, etc., and to prevent turbidity. This treatment deepens the color very much.

(b) DETECTION.

Place 50 cc of the extract to be examined in a glass dish and evaporate the alcohol on the water bath. When the alcohol is removed, make up to about the original volume with hot water. If alkali has not been used in the manufacture of the extract, the resins will appear as a flocculent red to brown residue. Acidify with acetic acid to free the resins from the bases, separating the whole of the resin and leaving a partly decolorized, clear, supernatant liquid after standing a short time. Collect the resin on a filter, wash with water, and reserve the filtrate for further tests.

Place a portion of the filter with the attached resins in a few cubic centimeters of dilute caustic potash. The resin is dissolved, giving a deep red solution; acidify, and the resin is thereby precipitated.

Dissolve a portion of the resin in alcohol; to one fraction add a few drops of ferric chlorid; no striking coloration is produced. To another portion add hydrochloric acid; again there is little change in color. In alcoholic solution most resins give color reactions with ferric chlorid or hydrochloric acid.

To a portion of the filtrate obtained above add a few drops of lead subacetate. The precipitate is so bulky as to almost solidify, due to the excessive amount of organic acids, gums, and other extractive matter. The filtrate from this precipitate is almost colorless.

Test another portion of the filtrate from the resin for tannin with a solution of gelatin. Tannin is present in varying but small quantities. It should not be present in great excess.

10. Detection and Determination of Methyl Alcohol.

Proceed as directed under "XV. Distilled Liquors," on page 99, section 12.

11. Test for Coloring Matter (Caramel).

(a) PRELIMINARY TEST.

If on shaking the bottle of vanilla the bubbles formed are of a bright caramel color and they keep this color until the very last are gone, it indicates presence of caramel. This difference is readily shown by comparison with known pure samples.

(b) LEAD ACETATE TEST.

The coloring matter present in vanilla or tonka extracts is almost completely removed when the dealcoholized extract is treated with a few cubic centimeters of basic lead acetate solution. When caramel is present, the filtrate and precipitate, if any, have the characteristic red-brown color of caramel.

LEMON EXTRACT.

1. Specific Gravity.

Determine as directed under "XIII. Wine," on page 83.

## 2. Alcohol.

Dilute 50 cc of the extract, measured at 15.6° C., to 200 cc, place the flask in a centrifuge and run until the oil separates in a clear layer at the top; then make up to the mark, using the lower meniscus of the oil. Pour the mixture into a dry Erlenmeyer flask containing 5 grams of light carbonate of magnesia, stopper, shake well, and filter quickly through a large, dry, folded filter. Determine the alcohol in 150 cc of the filtrate as directed under "XV. Distilled Liquors," page 95.

## 3. Glycerol.

Proceed as directed under "XIII. Wine," on page 83.

## 4. Lemon Oil.

### (a) BY POLARIZATION (MITCHELL).

Polarize the extract without dilution in a 200-mm tube at a temperature of 20° C., using the S. and H. sugar scale. Divide the reading by 3.2 and, in the absence of other optically active substances, the result will be the percentage of lemon oil by volume.

A small amount of cane sugar is occasionally present, being used to facilitate solution of the oil. In such cases determine it as directed under sucrose and correct the reading accordingly.

### (b) BY PRECIPITATION (MITCHELL).

Pipette 20 cc of the extract into a Babcock milk bottle; add 1 cc dilute hydrochloric acid (1:1); then add from 25 to 28 cc of water previously warmed at 60° C.; mix and let stand in water at 60° for 5 minutes; whirl in centrifuge for 5 minutes; fill with warm water to bring the oil into the graduated neck of the flask; repeat whirling for 2 minutes; stand the flask in water at 60° C. for a few minutes and read the per cent of oil by volume. In case oil of lemon is present in amounts over 2 per cent add to the percentage of oil found 0.4 per cent to correct for the oil retained in solution. If less than 2 per cent and more than 1 per cent is present, add 0.3 per cent for correction.

When the extract is made in accordance with the United States Pharmacopœia, the results by the two methods just given should agree within 0.2 per cent.

To obtain per cent by weight from per cent by volume, as found by either of the above methods, multiply the volume percentage by 0.86 and divide the result by the specific gravity of the original extract.

Negative results by the above methods are conclusive as to the absence of lemon oil. Positive results, however, should be confirmed by determining the physical constants of the precipitated oil.

## 5. Refraction of Precipitated Oil.

Determine the refractive index of the precipitated oil as directed under "XIX. Edible Oils and Fats," page 131.

Limonene and most commercial adulterants give a higher reading than lemon oil, with the exception of citronella aldehyde and oil of turpentine.<sup>a</sup>

<sup>a</sup> J. Amer. Chem. Soc., 1899, 21: 1132.



**6. Total Solids.**

Evaporate 10 cc of the sample on a water bath at a low temperature, and determine total solids as directed under "XIII. Wine," page 84, paragraph "5. Extract."

**7. Ash.**

Determine in the residue from 10 cc of the extract as directed under "VI. General Methods," page 38.

**8. Sucrose.**

Neutralize the normal weight of the extract, evaporate to dryness, wash several times with ether, dissolve in water, and determine as directed under "VI. General Methods," page 40.

**9. Methyl Alcohol.**

Make quantitative and qualitative determinations as directed under "XV. Distilled Liquors," on page 99, using the distillate from the determination of alcohol, paragraph 2, page 160.

**10. Detection of Coloring Matter.**

Proceed as directed under "XXVIII. Coloring Matters," page 190.

## XXIV. METHODS FOR THE ANALYSIS OF SPICES.

### CHEMICAL EXAMINATION.

#### 1. Preparation of Sample.

Grind the sample until it will pass through a sieve having round holes 1 mm in diameter. For the determination of starch in pepper, by the diastase method, reduce a portion of the sample to an impalpable powder by grinding it in a mortar.

#### 2. Moisture.

Dry 2 grams at 110° C. to constant weight. From the resulting loss in weight subtract the amount of volatile ether extract determined as in paragraph 9, page 163.

#### 3. Ash.

Determine as directed under "VI. General Methods," page 38. If calcium carbonate be present, care must be taken to burn the material, and also the residue after exhaustion with water, at a heat below redness, thus avoiding loss of the carbonic acid of the carbonate.

#### 4. Soluble Ash.

Determine as directed under "X. Saccharine Products," section 3 (c), page 68.

#### 5. Ash Insoluble in Acid.

Incinerate 2 grams of the sample as directed under section 3, boil with 25 cc of 10 per cent hydrochloric acid (sp. gr. 1.050) for five minutes, collect the insoluble matter in a gouch, wash with hot water, ignite, and weigh.

#### 6. Lime in Ash.

Determine as directed under "XXVI. Baking Powder," etc., on page 178.

#### 7. Nitrogen.

Determine as directed under "VI. General Methods," on page 5, except in the case of pepper, when the Gunning-Arnold method should be used, as follows:

##### GUNNING-ARNOLD METHOD.

(For black and white pepper.)

Owing to the presence of piperine the Gunning-Arnold method<sup>a</sup> must be used to determine nitrogen in both black and white pepper. Mix 1 gram of the

<sup>a</sup> Zts. anal. Chem., 1892, 31: 525; Conn. Agr. Exper. Stat. Rept., 1898, p. 190.

material in a 600 cc flask with 1 gram of copper sulphate, 1 gram of mercuric oxid, from 15 to 18 grams of potassium sulphate, and 25 cc of sulphuric acid. After heating gently until frothing ceases, boil the mixture for two to four hours. When nearly cool add about 300 cc of water, 50 cc of 4 per cent potassium sulphid solution, and sodium hydroxid solution to alkaline reaction. Distil into standard acid and titrate with standard alkali, as usual.

#### 8. Nitrogen in Nonvolatile Ether Extract—Winton, Ogden, and Mitchell Method.

(For black and white pepper.)

Extract 10 grams of pepper for twenty hours in continuous extraction apparatus with absolute ether, collecting the extract in a weighed flask of about 250 cc capacity. Evaporate the ether, dry first at 100° C., and finally to constant weight at 110° C. Determine the nitrogen in the weighed extract by the Gunning-Arnold method, digesting in the same flask used for the extraction. Calculate the parts of nitrogen in 100 parts of nonvolatile ether extract. Black pepper, owing to the presence of piperine, contains not less than 3.25 parts, and white pepper not less than 4 parts, of nitrogen. Linseed meal and other oily adulterants may contain about the same amount of ether extract as pepper, but this extract is practically free from nitrogen.

If desired, crude piperine may be calculated from the nitrogen by multiplying by 20.36.

#### 9. Ether Extract, Volatile and Nonvolatile.<sup>a</sup>

Extract 2 grams of the ground material for twenty hours in a continuous extraction apparatus with absolute ether. Transfer the ethereal solution to a tared capsule and allow to evaporate at room temperature. Let stand for eighteen hours over sulphuric acid, and weigh the total ether extract. Heat the extract gradually and then at 110° C. until the weight becomes constant. The loss is volatile oil; the residue, nonvolatile ether extract.

#### 10. Alcohol Extract.<sup>b</sup>

Place 2 grams of material in a 100 cc flask and fill to the mark with 95 per cent alcohol by volume. Stopper, shake at intervals of thirty minutes for eight hours, and allow to stand for sixteen additional hours without shaking. Filter the extract through a dry filter, evaporate 50 cc to dryness in a flat-bottomed dish on a water-bath, and heat to constant weight at 110° C.

#### 11. Copper Reducing Matters by Direct Inversion Calculated as Starch.

Extract 4 grams of the sample on a filter that will completely retain the smallest starch granules, with five successive portions of 10 cc of ether. After the ether has evaporated wash with 150 cc of 10 per cent (by volume) alcohol.

Since it is not possible to wash samples of Batavia cassia with water or dilute alcohol, owing to the formation of a glutinous mass which clogs the filter, for the sake of uniformity all preliminary washing is best omitted in determinations made on all varieties of cassia, as well as on cassia buds and cinnamon.

Carefully wash the residue from the paper into a 500 cc flask with 200 cc of

<sup>a</sup> Richardson, U. S. Dept. Agr., Division of Chemistry, Bul. 13, Part 2, p. 165.

<sup>b</sup> Conn. Agr. Exper. Stat. Rept., 1898, p. 187.

water, using a small wash bottle, and gently rubbing the paper with the tip of the finger.

Determine copper reducing material as directed under "VI. General Methods," section "8. Starch (a), Direct Acid Hydrolysis," page 53.

### 12. Starch.

Extract 4 grams of the finely pulverized material with ether and 10 per cent alcohol, as described in the preceding section, and determine starch by the diastase method, as directed under "VI. General Methods," on page 53.

### 13. Crude Fiber.

Determine as directed under "VI. General Methods," on page 56, except that the fiber is filtered and weighed on a paper rather than on a gooch, since the latter is liable to clog, rendering filtration impossible.

### 14. Tannin.

(For cloves and allspice.)

Extract 2 grams of material for twenty hours with absolute ether. Boil the residue for two hours with 300 cc of water, cool, make up to 500 cc, and filter. Measure 25 cc of this infusion into a flask of about 1,200 cc capacity, add 20 cc of indigo solution and 750 cc of distilled water, and proceed as directed under "XIII. Wine," page 88.

Ten cubic centimeters of tenth-normal oxalic-acid solution are equivalent to 0.06232 gram of quercitannic acid, or 0.008 gram of oxygen absorbed.

### 15. Cold-Water Extract.

(For ginger.)

Place 4 grams in a graduated 200 cc flask, add water to the mark, shake at half-hour intervals during 8 hours and let stand 16 additional hours without shaking. Filter and evaporate 50 cc to dryness in a flat-bottomed metal dish. Dry to constant weight at 100° C.

### 16. Total Sulphur.

(For mustard.)

Determine as directed under "III. Inorganic Plant Constituents," page 23.

## MICROSCOPIC EXAMINATION.

The microscope is the most valuable means of detecting adulterants of vegetable origin in spices, as it usually discloses the particular adulterant present, even when in small amount. The analyst who undertakes this work should have a general knowledge of vegetable histology and microscopic manipulation, and should be thoroughly familiar with the microscopic appearance of the spices and spice adulterants. He should have at his command the

standard works on these subjects, and also a set of standard samples of all the materials likely to be encountered.<sup>a</sup>

(a) APPARATUS.

- (1) Dissecting microscope or hand lens.
- (2) Compound microscope provided with  $\frac{2}{3}$  and  $\frac{1}{3}$  inch objective, 1 and 2 inch oculars, double nosepiece, eyepiece micrometer, and polarizing apparatus.
- (3) A series of sieves with meshes ranging from 0.2 to 2 mm.
- (4) Slides, cover glasses, needles, scalpels, forceps, etc.

(b) MICRO-REAGENTS.

Of the numerous reagents employed in histological work the following are most useful in spice examination:

- (1) Distilled water.
- (2) Glycerin, pure and diluted with equal volume of water.
- (3) Absolute alcohol.
- (4) Ether.
- (5) Ammonium hydroxid.
- (6) Potassium hydroxid (5 per cent).
- (7) Chloral hydrate (8 parts in 5 parts of water).
- (8) Schultze's macerating mixture.—Crystallized potassium chlorate mixed with nitric acid as needed.
- (9) Iodin in potassium iodid.—0.05 gram iodine, 0.2 gram potassium iodid, and 15 cc water.
- (10) Chlorzinc iodine.—Dissolve 100 grams of zinc chlorid in 60 cc of water and to this add 20 grams of potassium iodid and 0.5 gram of iodine crystals. A few crystals of iodine should be left in the bottle to insure saturation. Allow the solution to stand a few hours before using. Chlorzinc iodine prepared in this manner will keep for months. If the color developed in the tissue is too deep a blue a very slight dilution of the reagent is advisable.
- (11) Millon's reagent.—Dissolve metallic mercury in its weight of concentrated nitric acid, add an equal volume of water, and decant off the clear liquid as soon as the precipitate settles.
- (12) Ferric acetate or chlorid solution.
- (13) Alkanna tincture diluted with an equal bulk of water.
- (14) Aqueous solution of safranin.
- (15) Hydrochloric acid (10 per cent).
- (16) Acetic acid.

(c) PREPARATION OF THE SAMPLE.

Reduce one portion to a fine powder in a mortar. Separate another portion into several grades of fineness in sieves of different mesh or by jarring on a sheet of paper. In the coarser grades fragments of a suspicious nature may often be seen with the naked eye or under a simple microscope; these should be picked out for subsequent examination under the compound microscope.

<sup>a</sup>The following works are especially recommended: Winton, *Microscopy of Vegetable Foods*, 1906, New York; Moeller, *Mikroskopie der Nahrungs- und Genussmittel aus dem Pflanzenreiche*, 1905, Berlin; Vogl, *Die wichtigsten vegetabilischen Nahrungs- und Genussmittel*, 1899, Berlin; Tschirch und Oesterle, *Anatomischer Atlas der Pharmakognosie und Nahrungsmittelkunde*, 1900, Leipzig.

## (d) EXAMINATION.

Mount a small quantity of the ground sample in water and examine under the compound microscope with both ordinary and polarized light. This gives a general insight into the nature of the material and serves for the detection and identification of starch granules and various tissues.

Draw a small drop of iodine solution into the same preparation by means of a piece of filter paper placed on the opposite edge of the cover glass and examine. Starch granules will be colored blue or blue-black, cellulose yellow, and proteids either brown or yellow.

In the manner described draw a little potassium hydroxide solution under the cover glass and examine once again. This treatment gelatinizes the starch granules, dissolves the proteids, saponifies the fats, and in other ways clears the preparation. It also imparts to tannins a reddish color.

If treatment with potash does not clear the tissues satisfactorily, treat a fresh portion for some hours with chloral hydrate solution.

Examine also the crude fiber obtained in the chemical analysis, as in this material the stone cells and other tissues are beautifully distinct.<sup>a</sup>

To isolate stone cells, bast fibers, and other thick-walled cells macerate a portion of the sample in Schultze's liquid, using such proportion of potassium chlorate and nitric acid and heating for such a time as secures the desired results. Powdered charcoal and charred shells resist the bleaching action of potash, chloral hydrate, and Schultze's liquid, the fragments after, as before the treatment, being black and opaque.

If it is desired to distinguish cellulose from infiltrated substances (lignin, suberin, etc.), add to a water mount freshly prepared chlorzinc iodine, which colors the former blue and the latter yellow.

As a test for proteids, cautiously warm, on a slide, with a drop of freshly prepared Millon's reagent. The proteids are partially disorganized, taking on gradually a brick-red color. If it is desirable to study the form of the aleurone (protein) granules, which in some plants are quite as characteristic as starch granules, prepare a mount in pure glycerin or oil.

To distinguish fats, oils, essential oils, and resins from other cell contents, treat for an hour with alkanna tincture diluted with an equal bulk of water, which imparts to these substances a deep red color, or treat with ether, which dissolves them. Treat also with alcohol, which dissolves the essential oils and resins, but does not perceptibly affect the fats and oils.

In testing for tannins and tissues impregnated with those substances, add ferric acetate or chloride solution. Both of these reagents give with tannins a green or blue color, but the former acts more slowly and is to be preferred.

Crystals of calcium oxalate are recognized by their characteristic forms and their deportment with polarized light. To distinguish calcium oxalate from calcium carbonate, treat with acetic acid, which does not affect the former, but dissolves the latter with effervescence. Both are soluble in hydrochloric acid.

Other special reagents play a subordinate part in the microscopic examination of spices, the chief factor being a thorough understanding of the size, shape, color, and other characteristics of the histological elements, which can be learned only by experience.

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<sup>a</sup> Winton, Conn. Agr. Exper. Stat. Rept., 1896, p. 34.

## **XXV. METHODS FOR THE ANALYSIS OF CONDIMENTS OTHER THAN SPICES.**

### **PREPARED MUSTARD.**

#### **1. Preparation of Sample.**

The solid portion of the material is commonly in a finely divided condition and does not require grinding, but as it tends to settle, leaving a more or less clear liquid on the surface, thorough mixing is absolutely essential. This may be accomplished by stirring well immediately before removing each portion for analysis.

#### **2. Solids.**

Dry 5 grams as directed under "VI. General Methods," on page 38.

#### **3. Ash.**

Burn the dry residue, obtained in the determination of moisture, as directed under "VI. General Methods," on page 38.

#### **4. Salt.**

Determine chlorine in the ash as directed under "III. Inorganic Plant Constituents," on page 23.

#### **5. Ether Extract.**

In a capsule place 10 grams of the material, about 30 grams of sand, and a short stirring rod. Heat on a water bath or in a water oven. Grind until all the lumps are broken up, and determine the ether extract as directed under "VI. General Methods," on page 39.

#### **6. Protein.**

Determine the nitrogen by the Kjeldahl or Gunning method as directed on page 5, under "I. Fertilizers."

#### **7. Acidity.**

Weigh 10 grams into a 200 cc graduated flask, make up to the mark with water, shake, filter through a dry paper and determine the acidity in 100 cc by titration with tenth-normal alkali, using phenolphthalein as indicator. State the results as acetic acid. One cubic centimeter of tenth-normal alkali is equivalent to 0.0060 gram of acetic acid.

#### **8. Copper-Reducing Matters Calculated as Starch.**

Proceed as directed under "VI. General Methods" for the determination of starch by acid hydrolysis, on page 53, except that 10 grams of the material are

treated directly with 200 cc of water and 20 cc of 25 per cent hydrochloric acid without previous washing or extraction, and the solution is made up to 250 cc before filtering and drawing off the aliquot.

#### 9. Crude Fiber.

Weigh 8 grams of the material (equivalent to about 2 grams of dry matter) directly into an Erlenmeyer flask and proceed as directed under "XXIV. Spices," on page 164, except that care is taken to add at first only a small amount of 1.25 per cent acid or alkali, and shake thoroughly until all lumps are broken up. If this precaution is not taken, the lumps will resist the action of the acid or alkali and the results will be high. Extraction of the fat previous to the treatment is impracticable, as this necessitates preliminary drying, after which the material forms a horny mass.

#### 10. Detection of Coloring Matter.

Proceed as directed under "XXVIII. Coloring Matter," on page 190.

#### 11. Detection of Preservatives.

Proceed as directed under "XXVII. Food Preservatives," on page 179.



## XXVI. METHODS FOR THE ANALYSIS OF BAKING POWDER AND BAKING CHEMICALS.—PROVISIONAL.

All the processes hereafter described, except determination of acidity, may be employed in the analysis of baking powders, and all the processes, except determination of carbonic acid, in the analysis of cream of tartar and its substitutes. The sample under examination is entirely removed from the package, carefully mixed, and passed through a sieve without grinding.

### 1. Total Carbon Dioxid.

Make the determination by the absorption method, employing any apparatus which gives accurate results when checked with pure calcite. Whatever apparatus is chosen, the tubes and materials used for absorbing and drying the carbon dioxid may be varied according to the preference of the analyst. According to the amount of absorbent employed vary the weight of sodium or calcium carbonate from 0.25 to 1 gram, and use about twice as much baking powder. The corrections for temperature and pressure given with the Heidenhain apparatus may ordinarily be disregarded.

#### (a) METHOD USING KNORR'S APPARATUS.

##### (1) DESCRIPTION OF APPARATUS.

This apparatus (fig. 9) has only ground-glass joints, and may be quickly made ready for use or taken to pieces and packed away. On the other hand, it is inflexible and must be carefully handled, and has the additional disadvantage that broken parts can not readily be replaced. Therefore it is of more value for occasional determinations than for a long series.<sup>a</sup>

##### (2) PREPARATION OF REAGENTS.

(1) *The potassium hydroxid* solution usually employed for absorbing carbon dioxid has a specific gravity of about 1.27. Many analysts, however, prefer a solution having a specific gravity of about 1.55.

(2) *The calcium chlorid and soda lime* employed should be finely granulated and freed from dust with a sieve.

##### (3) DETERMINATION.

Place some of the baking powder in a perfectly dry distilling flask. Close the flask with a stopper carrying the tube connecting with the absorption apparatus and also with the funnel tube. Weigh the tubes in which the carbon

<sup>a</sup> The small calcium chlorid tube shown in the cut attached to the potash bulb F is usually replaced by a second Liebig bulb with sulphuric acid. Better results are obtained if the same drying tubes are used before and after the potash bulb. Many analysts prefer to replace the bulb F and attached calcium chlorid tube by two U-tubes filled with sifted soda lime. When the second tube shows a material increase in weight it is placed first, and the first tube refilled and placed in the second position.

dioxid is to be absorbed and attach them to the apparatus. In case two Liebig bulbs are employed, one for potassium hydroxid and the other for sulphuric acid to absorb the moisture given up by the potassium hydroxid solution, weigh them separately. If two soda-lime tubes are employed it will be found advantageous to weigh them separately and fill the first tube anew when the second tube begins to increase in weight materially. Nearly fill tube B with hydrochloric acid (sp. gr. 1.1) and place the guard tube C in position. Then start the aspirator at such a rate that the air passes through the Liebig bulb at about the rate of two bubbles per second. Open the stopper of the funnel and allow

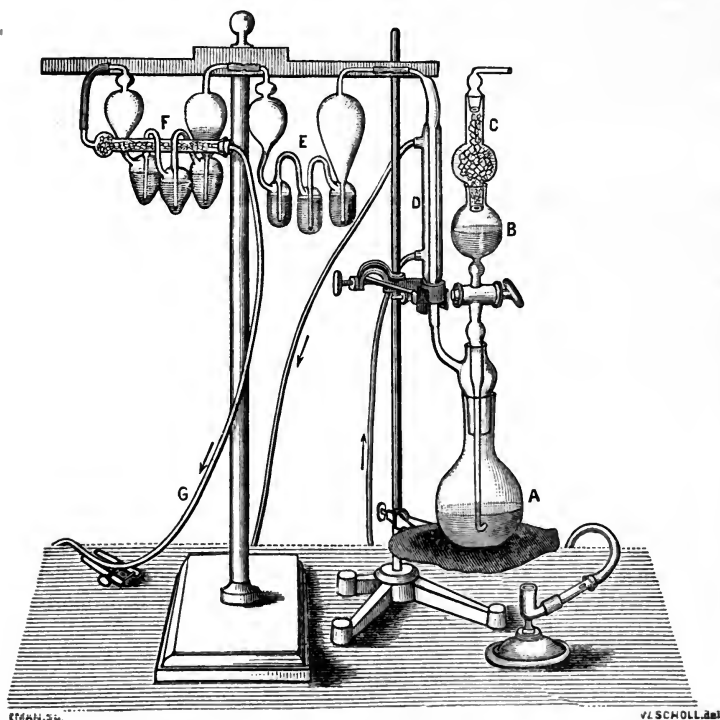


FIG. 9.—Knorr's apparatus for the determination of carbon dioxid; A, distilling flask fitted to condenser by a ground-glass stopper. B, Reservoir containing acid. C, Soda-lime tube fitted to acid reservoir by a ground-glass stopper. D, Condenser. E, Liebig bulb filled with sulphuric acid. F, Liebig bulb with a solution of potassium hydroxid for the absorption of carbon dioxid and followed by a calcium-chlorid tube. An additional guard tube filled with soda lime should follow the tube F, though not shown in the cut.

the acid to run slowly into the flask, care being taken that the evolution of gas shall be so gradual as not to materially increase the current through the Liebig bulb. After all the acid has been introduced continue the aspiration and gradually heat the contents of the flask to boiling, the bulb in tube B being closed. While the flask is being heated the aspirator tube may be removed, although many analysts prefer when using ground-glass joints to aspirate during the entire operation. Continue the boiling for a few minutes after the water has begun to condense in D, then remove the flame, open the valve in tube B, and allow the apparatus to cool with continued aspiration. Remove the absorption tubes and weigh. The increase in weight is due to carbon dioxid.

## (b) METHOD USING HEIDENHAIN'S APPARATUS.

## (1) DESCRIPTION OF APPARATUS.

This apparatus was originated by G. J. Mulder, recommended and improved by Kolbe, Stolba, and Fresenius,<sup>a</sup> and modified by H. Heidenhain.<sup>b</sup> As shown in fig. 10, drawn on a scale of 1:12, it consists of—

A. A cylinder filled with soda lime to free the air from carbon dioxide. A thick layer of cotton prevents soda-lime dust from being carried over.

B. Glass cock to regulate the air current, which finds resistance at C.

C. A capillary contraction.

D. Funnel tube of peculiar shape. The funnel is cylindrical, three-fourths of an inch wide and 4 inches long, and is reduced to half its width at the bottom, so as to make a neck for a perforated rubber stopper.

E. A glass tube is tightly fitted into the perforated rubber stopper, allowing the stopper to be taken out and replaced by the glass tube.

F. Evolution flask, ordinarily of 150 cc capacity, for foaming liquids of 300 cc capacity.

G. Return condenser, simply a glass tube of one-fourth of an inch bore, around which a small lead pipe is wound. The tube following the condenser contains a few pieces of calcium chlorid to retain the bulk of the moisture. It is refilled when contents are liquefied.

H. U tube filled with coarse calcium chlorid.

K. Filled at I with a 3-inch long column of pumice stone impregnated with copper sulphate completely dehydrated at 150° C. The remainder of the tube is filled with fine calcium chlorid.

L. Cock to close the apparatus when not in use.

M. First absorption tube about one-half inch in diameter and 5 inches long, filled mainly with soda lime, with a little calcium chlorid at the side at which the air current enters.

N. Second absorption tube of same size as M, filled half with soda lime and half with calcium chlorid. Place the side containing calcium chlorid toward the end of the apparatus where the air current leaves.

O. Guard tube containing calcium chlorid toward N and soda lime toward P.

P. Indicator tube trapped with glycerin.

R. Safety bottle to receive water which may be sucked back from the aspirator.

S. The aspirator, which is a Mariette's bottle of about 4 liters capacity.

## (2) PREPARATION OF REAGENTS.

Use calcium chlorid dehydrated at 200° C., not fused. Grind it coarsely in a coffee mill and sift through No. 18 wire gauze to remove the extremely coarse and through No. 30 wire gauze to remove the very fine. Prepare a large quantity of such calcium chlorid at the beginning and use this for the tubes K, M, and N. The reason for this is that the current of air must leave the weighed tubes with the same content of moisture as it entered them, which only can be attained if the absorbent in K and N is of the same nature and quality.

Grind and sift the soda lime<sup>c</sup> for the weighed tubes in the same way. It

<sup>a</sup> Fresenius, *Quantitative Analysis*, 1: 449; 2: 308. German edition.

<sup>b</sup> J. Amer. Chem. Soc., 1896, 18: 1.

<sup>c</sup> An excellent method for the preparation of soda lime is given by Benedict and Tower, J. Amer. Chem. Soc., 1899, 21: 396.

should not be too dry, as it must not absorb moisture to a higher degree than calcium chlorid. The tubes M and N should hold about 20 grams, making M's capacity for carbon dioxide almost 1 gram and N's capacity for moisture 0.2

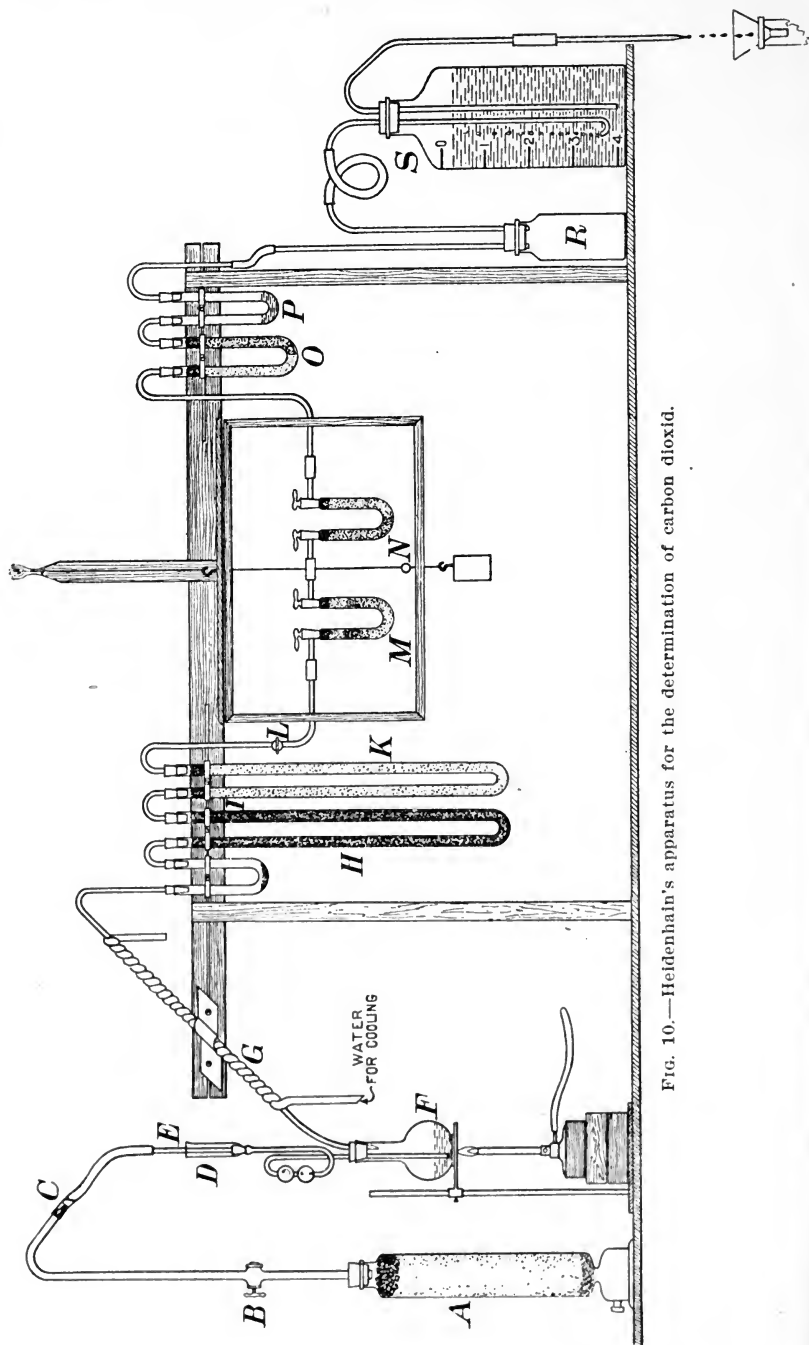


Fig. 10.—Heidenhain's apparatus for the determination of carbon dioxide.

gram. M should be refilled when its weight has increased 0.75 gram and N after an increase of 0.1 gram in weight.

Use best rubber for all connections, applying a trace of castor oil as lubricator. For connections of the weighed tubes use rubber tubing boiled in weak lye, washed and dried. Apply also a little castor oil, which is thoroughly wiped off again before connecting the tubing.

Before using the apparatus fill H and K with carbon dioxide in order to saturate the alkalinity of the calcium chloride, and exhaust after several hours.

### (3) DETERMINATION.

Weigh the tubes M and N, taking precaution that they are of the same temperature as the air in the balance room. Shortly before weighing open the tubes for a moment to allow equalization of air. Note thermometer and barometer. Connect tubes with the apparatus and make sure that all joints are tight by closing A at the bottom, opening all cocks, starting the aspirator, and observing P, in which the liquid must soon come to a standstill. Then disconnect the aspirator, close B, remove F, put in the substance (use about 1 gram of sodium carbonate or calcium carbonate, or about 2 grams of baking powder), connect F, and start the cooler. Introduce acid and water through D, lifting E slightly, and allowing only small quantities of the acid and water to enter at the time. Use only carbon dioxide free water. Light the burner, heat to boiling, and reduce the flame to keep the liquid just at the boiling point. If no more air passes P, start the aspiration. When water stops running, open B carefully and adjust the outflow of the aspirator by raising or lowering the syphon to half the safe speed.

(In order to find the allowable rapidity of the air current, proceed as follows: Charge the apparatus exactly as for an analysis, leaving out the carbonate. Begin to aspirate at the rate of about 50 cc per minute. After 2 liters have been aspirated weigh the tubes. If they have lost in weight, repeat the experiment with 40 cc per minute, and so on until the weight of the tubes remains constant. If the work has been done with due precaution, the first tube must have lost just as much as the second has gained. Do not exceed the safe speed thus found.)

After M has become cool increase the current to full safe speed and aspirate altogether 3 liters, continuing boiling to the end of aspiration. After the tubes have assumed the temperature of the balance room open for a moment, weigh, note again thermometer and barometer, and apply correction according to the following formulæ:

$$-(A^2-A^1) \times T \text{ and } +(B^2-B^1) \times B$$

In which

$A^1$  is the temperature at first weighing in °C,

$A^2$  is the temperature at second weighing in °C,

$B^1$  is the air pressure at first weighing in mm,

$B^2$  is the air pressure at second weighing in mm,

and T and B are constants found as follows:

G=weight of empty tubes.

F=weight of fillings.

The specific gravity of glass=2.7.

The specific gravity of filling=2.0.

The specific gravity of brass=8.5.

Change of weight of 1 cc air with  $1^\circ=0.0000039$  gram.

Change of weight of 1 cc air with 1 mm pressure=0.0000015 gram.

From above follows:

$$\text{Volume of tubes and fillings} = \frac{G}{2.7} + \frac{F}{2.0}.$$

$$\text{Volume of brass weights} = \frac{G+F}{8.5}.$$

and

$$\frac{G}{2.7} + \frac{F}{2.0} - \frac{G+F}{8.5} = V,$$

representing the differential volume affected by temperature and pressure and being a constant for the tubes. Now,

$$T = V \times 0.0000039 \text{ gram, and}$$

$$B = V \times 0.0000015 \text{ gram.}$$

Observe that rise of temperature makes the air lighter, consequently the tubes heavier. Therefore the correction must be negative. On the other hand, increased pressure has the opposite effect, making the correction positive.

Example:

$$G=80. \quad F=40, \text{ from which follows}$$

$$V=35.5, \text{ and}$$

$$T=0.00014 \text{ gram, and}$$

$$B=0.00005 \text{ gram.}$$

Now, if—

$$A^1=25^\circ,$$

$$A^2=270^\circ,$$

$$B^3=759 \text{ mm,}$$

$$B^4=756 \text{ mm,}$$

then the correction for temperature will be—

$$-0.00028 \text{ gram,}$$

and for air pressure—

$$-0.00015 \text{ gram,}$$

making a total of—

$$-0.00043 \text{ gram.}$$

## 2. Residual Carbon Dioxid.<sup>a</sup>

Weigh 2 grams of baking powder into a flask suitable for the subsequent determination of carbonic acid, add 20 cc of cold water, and allow to stand 20 minutes. Place the flask in a metal drying cell surrounded by boiling water and heat, with occasional shaking, for 20 minutes.

To complete the reaction and drive off the least traces of gas from the semi-solid mass, heat quickly to boiling and boil for 1 minute. Aspirate until the air in the flask is thoroughly changed, and determine the residual carbon dioxid by absorption, as described, under total carbonic acid.

The process described,<sup>a</sup> based on the methods of McGill<sup>b</sup> and Catlin,<sup>c</sup> imitates as far as practicable the conditions encountered in baking but in such a manner that concordant results may be readily obtained on the same sample and comparable results on different samples.

<sup>a</sup> Conn. Agr. Exper. Stat., Rept., 1900, p. 169.

<sup>b</sup> Laboratory of the Inland Revenue Department, Ottawa, Canada, Bul. 68, p. 31.

<sup>c</sup> Baking Powders: A Treatise on their Character, Method for Determination of the Values, etc., p. 20.

### 3. Available Carbon Dioxid.

Subtract the residual carbon dioxid from the total.

### 4. Acidity.

(For cream of tartar and its substitutes.)

Dissolve 1 gram of the material in hot water and titrate with standard fifth-normal potassium hydroxid solution, using phenolphthalein as indicator.

### 5. Detection of Tartaric Acid, Free or Combined "—Wolff Method.

(Applicable in presence of phosphates.)

Shake repeatedly about 5 grams of the sample with about 250 cc of cold water in a flask and allow the insoluble portion to subside. Decant the solution through a filter and evaporate the filtrate to dryness. To the dry powdered residue add a few drops of a 1 per cent solution of resorcin and about 3 cc of strong sulphuric acid. Heat slowly. A rose-red color indicates tartaric acid, the color being discharged on dilution with water.

### 6. Detection of Free Tartaric Acid.

Extract 5 grams of the powder with absolute alcohol and evaporate the alcohol from the extract. Dissolve the residue in dilute ammonium hydroxid, transfer to a test tube, add a good-sized crystal of silver nitrate, and heat gently. Tartaric acid is indicated by the formation of a silver mirror. If desired, the absolute alcohol extract may be tested by the Wolff method, as described under paragraph 5.

### 7. Total Tartaric Acid—Goldenberg-Geromont-Heidenhain Method.

(Applicable only in the absence of aluminum salts, calcium salts, and phosphates.)

Into a shallow porcelain dish, 6 inches in diameter, weigh out 2 grams of the material and sufficient potassium carbonate to combine with all tartaric acid not in the form of potassium bitartrate. Mix thoroughly with 15 cc of cold water and add 5 cc of 99 per cent acetic acid. Stir for half a minute with a glass rod bent near the end. Add 100 cc of 95 per cent alcohol, stir violently for 5 minutes, and allow to settle at least 30 minutes. Filter on a gooch crucible with a thin layer of paper pulp, and wash with 95 per cent alcohol until 2 cc of the filtrate do not change the color of litmus tincture diluted with water. Place the precipitate in a small casserole, dissolve in 50 cc of hot water and add standard fifth-normal potassium hydroxid solution, leaving it still strongly acid. Boil for 1 minute. Finish the titration, using phenolphthalein as indicator and correct the reading by adding 0.2 cc. One cubic centimeter of fifth-normal potassium hydroxid solution is equivalent to 0.026406 gram of tartaric anhydrid, 0.03001 gram of tartaric acid, or 0.03763 gram of potassium bitartrate.

The standard of the potassium hydroxid solution should be fixed by pure dry potassium bitartrate.

The accuracy of this method is indicated by the agreement of the percentages of potassium bitartrate in cream of tartar powders containing no free tartaric acid, obtained by calculation from the tartaric acid, with those obtained by calculation from the potassium oxid.<sup>a</sup>

## 8. Starch.

### (a) DIRECT INVERSION METHOD.

(For all baking chemicals free from lime.)

Weigh 5 grams of the powder into a graduated 500-cc flask. Convert into dextrose by the modified Sachsse method ("VI. General Methods," page 53), and determine the reducing power by the Allihn method (page 49).

### (b) INDIRECT METHOD.<sup>b</sup>

(For phosphate, alum phosphate, and all other baking powders containing lime.)

Mix 5 grams of the powder in a graduated 500-cc flask with 200 cc of 3 per cent hydrochloric acid, and allow the mixture to stand for 1 hour, with frequent shaking. Filter on an S. & S. No. 575, 11 cm, hardened filter, taking care that a clear filtrate is obtained. Rinse the flask once without attempting to remove all the starch, and wash the paper twice with cold water. Carefully wash the starch from the paper back into the flask, with 200 cc of water, using a small wash bottle. Add 20 cc of 25 per cent hydrochloric acid and proceed according to the Sachsse method. Determine reducing power by Allihn's method.

The treatment with 3 per cent hydrochloric acid, without dissolving the starch, effectually removes the lime, which otherwise would precipitate as tartrate in the alkaline copper solution.

### (c) MODIFIED MCGILL METHOD.

Digest 1 gram of the powder with 150 cc of 3 per cent hydrochloric acid for 24 hours at room temperature, with occasional shaking. Filter on a gooch crucible, wash thoroughly with cold water and, finally, once with alcohol, and once with ether. Dry at 110° C. (4 hours is usually sufficient), cool, and weigh. Burn off the starch, weigh again, and determine by difference.

The results by this method on cream of tartar powders and tartaric acid powders agree closely with those obtained by copper reduction. On phosphate, alum, and alum-phosphate powders the results are usually satisfactory, but in some instances they may be over 2 per cent too high.

## 9. Potassium Bitartrate.

If, as is usually the case, no other potassium salt but the bitartrate is present, multiply the percentage of total potash determined as directed under section 12 (d) on page 178, by 3.9936.

<sup>a</sup> Conn. Agr. Exper. Stat. Rept., 1900, p. 180.

<sup>b</sup> Winton, Conn. Agr. Exper. Stat. Rept., 1900, 175.



### 10. Free Tartaric Acid.

Calculate the percentage of tartaric anhydrid combined with the potash as bitartrate, if any, and subtract this from the percentage of total tartaric anhydrid. The difference is the tartaric anhydrid originally added as the free acid, although if the sample has been kept for a long time or has been improperly stored a portion of all of this acid may exist at the time of analysis as the sodium salt resulting from the reaction in the can with the sodium bicarbonate. Multiply by 1.1365 to obtain the percentage of tartaric acid.

### 11. Detection of Alum in Presence of Phosphates.<sup>a</sup>

#### (a) IN BAKING POWDER.

Burn to an ash about 2 grams of the sample in a platinum dish. Extract with boiling water and filter. Add to the filtrate a few drops of ammonium chlorid solution. A flocculent precipitate indicates alum.

#### (b) IN CREAM OF TARTAR.

Mix about 1 gram of the sample with an equal quantity of sodium carbonate, burn to an ash, and proceed as in (a).

### 12. Examination of Ash.<sup>b</sup>

#### (a) INSOLUBLE ASH AND PREPARATION OF SOLUTION.

Char 5 grams in a platinum dish at a heat below redness. Boil the carbonaceous mass with dilute hydrochloric acid, filter into a graduated 500-cc flask, and wash with hot water. Return the residue, together with the paper, to the platinum dish and burn to a white ash. Boil again with hydrochloric acid, filter, wash, unite the two filtrates, and dilute to 500 cc.

Incinerate the residue after the last filtration for the determination of ash insoluble in acid.

#### (b) IRON AND ALUMINA.

Draw an aliquot of 100 cc and separate silica, if necessary. Mix the solution with sodium-phosphate solution in excess of what is required to form normal aluminum phosphate. Add ammonium hydroxid until a precipitate remains on stirring, then hydrochloric acid drop by drop until the precipitate dissolves. Heat the solution to about 50° C., mix with a considerable excess of 50 per cent ammonium acetate solution and 4 cc of 80 per cent acetic acid.

As soon as the precipitate of aluminum phosphate, mixed with iron phosphate, has settled, collect on a filter, wash with hot water, ignite, and weigh.

Fuse the mixed phosphates with 10 parts of sodium carbonate, dissolve in dilute sulphuric acid, reduce with zinc, and determine the iron by the volumetric permanganate method. In the same solution determine the phosphoric acid. To obtain the weight of  $\text{Al}_2\text{O}_3$ , subtract the sums of the weights of  $\text{Fe}_2\text{O}_3$  and  $\text{P}_2\text{O}_5$  from the weight of the mixed phosphates.

<sup>a</sup> Thirty-first Ann Rept. Mass. State Board of Health, 1899, p. 638.

<sup>b</sup> Conn. Agr. Exper. Stat. Rept., 1900, p. 175.

## (c) LIME.

Heat the filtrate from the mixed phosphates, which is acid with acetic acid, to 50° C. and precipitate with ammonium oxalate. Filter, wash, ignite over a Bunsen burner, and finally convert into oxid by heating over a blast lamp.

## (d) POTASH AND SODA.

Evaporate an aliquot of the solution, prepared as described, nearly to dryness to remove the excess of hydrochloric acid, dilute, and heat to boiling. While still boiling add barium chlorid solution as long as a precipitate forms and enough barium hydrate to make the liquid strongly alkaline. As soon as the precipitate has settled, filter and wash with hot water, heat the filtrate to boiling, add sufficient ammonium carbonate solution (1 part ammonium carbonate in 5 parts of 2 per cent ammonium water) to precipitate all the barium, filter, and wash with hot water. Evaporate the filtrate to dryness, ignite below redness to remove ammonium salts. Add to the residue a little water and a few drops of ammonium carbonate solution. Filter into a tared platinum dish, evaporate, ignite below redness, and weigh the mixed potassium and sodium chlorids.

Determine the potash as potassium platinichlorid, using the factors 0.1941 for  $K_2O$  and 0.3071 for  $KCl$ .

## 13. Phosphoric Acid.

Mix 5 grams of the material with a little magnesium-nitrate solution, dry, ignite, and dissolve in hydrochloric acid. In an aliquot of the solution determine phosphoric acid as magnesium pyrophosphate by the molybdate method as directed under "I. Fertilizers," page 4.

## 14. Sulphuric Acid.

Boil 5 grams of the powder gently for 1½ hours with a mixture of 300 cc of water and 15 cc of concentrated hydrochloric acid. Dilute to 500 cc, draw off an aliquot portion of 100 cc, dilute considerably, precipitate with barium chlorid, filter through a gooch, ignite, and weigh. Direct solution of the material without burning off the organic matter was proposed by Crampton.<sup>a</sup> The dextrose formed by the action of the acid on the starch of baking powders does not interfere with the accuracy of the process.<sup>b</sup>

## 15. Ammonia.

Ammonia alum is often an ingredient of cream of tartar substitutes and baking powders, and ammonium carbonate is occasionally present in baking powders. Determine ammonia by distillation with caustic soda into standard acid and titration.

<sup>a</sup> U. S. Dept. Agr., Division of Chemistry, Bul. 13, Part 5, p. 596.

<sup>b</sup> Conn. Agr. Exper. Stat. Rept., 1900, p. 179.

## XXVII. METHODS FOR THE DETECTION AND DETERMINATION OF FOOD PRESERVATIVES.—PROVISIONAL.

### 1. Salicylic Acid.

A small amount of salicylic acid occurs naturally in many fruits, and not more than 50 grams should be used for its qualitative detection in the examination of foods. A reaction obtained with this amount is due to added salicylic acid. The method described below is intended for the quantitative determination of salicylic acid. If only a qualitative determination be desired, many of the details may be omitted.

If the material be a solid or semisolid, macerate the sample in a mortar with water made slightly alkaline and strain through a cotton bag or separate by means of a centrifuge. If preferred, macerate from 200 to 300 grams with about 400 cc of water and use aliquots of the filtrate for the determination of preservatives.

In quantitative work place the macerated mass in a graduated flask, make up to a definite volume with water, and shake from time to time until solution is complete. Then strain as directed above and use an aliquot of the filtrate for extraction.

Extract in a separatory funnel 100 cc of the sample or of the aqueous solution prepared from the sample as described above with a sufficient amount of sulphuric ether<sup>a</sup> to prevent emulsion after the addition of 2 or 3 cc of dilute (1-3) sulphuric acid. Separate the clear aqueous solution, and if any emulsion is present give the separatory funnel a quick, vigorous shake and allow to settle again. If the emulsion is not broken up in this way, it may be accomplished

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<sup>a</sup> If the nature of the substance is such that extraction with organic solvents is not practicable, as in the case of the presence of a large amount of fat, the salicylic acid may first be separated by distillation. In such cases acidify the macerated material with phosphoric acid and transfer to a distilling flask with a very short neck and wide mouth. An Erlenmeyer flask with inside diameter of mouth of  $1\frac{1}{4}$  inches is a good shape. The tube connecting the flask with condenser should be very short, with an inside diameter of not less than three-eighths of an inch.

Conduct steam through a small tube passing through the stopper and dipping deeply into the material in the flask. The distillation of the salicylic acid is facilitated by submerging the distilling flask almost to the stopper in an oil bath and distilling with the temperature of the oil at from  $120^{\circ}$  to  $130^{\circ}$  C., or by adding about 20 grams of sodium chlorid to the contents of the flask for each 100 cc of the substance, to raise the boiling point. Care must be taken not to let the contents of the flask get too low, as the heat will decompose the organic matter.

Collect at least 600 cc of the distillate and continue the distillation until the last 200 cc gives no color on the addition of a drop of ferric solution. The distilling apparatus should in all cases be tested with known amounts of salicylic acid in order to determine the amount of distillate necessary to carry over a definite weight of salicylic acid.

It is sometimes practicable to determine the salicylic acid directly in the distillate by the colorimetric method with ferric chlorid given above. If the mineral acid used in the distillation be carried over mechanically, however, the accuracy of the method is greatly impaired. Salicylic acid may be recovered from the distillate after making alkaline and evaporating, if desired, by extraction with ether and estimating colorimetrically as directed above.

by means of a centrifuge or by adding 10 or 15 cc of low boiling point gasoline or petroleum ether and shaking again.

Separate the clear, aqueous portion obtained from the emulsion and add it to the first aqueous portion separated. Then pour the ether into another separatory funnel, care being taken that none of the aqueous portion is left with the ether. Return the aqueous portion to the separatory funnel and again extract with ether, following the same procedure as before. Repeat this operation twice again, four separate extractions with ether being made in all.

In case of special difficulty in breaking up the emulsion in any of the extractions a small amount of ether may be allowed to remain with the aqueous portion rather than the reverse, as it is removed in successive extractions. Wash the combined ether extracts by shaking in a separatory funnel with one-tenth their volume of water (using, however, not less than 20 cc of water at each washing). Care must be taken at each washing to separate the aqueous portion completely from the ether, but none of the ether should be allowed to run into the wash water.

Distil slowly the greater part of the ether, transfer the remainder to a porcelain dish, and allow to evaporate spontaneously. Thoroughly dry in a vacuum desiccator <sup>a</sup> over sulphuric acid, extract the dry residue with ten portions of 10 or 15 cc each of carbon bisulphid or low boiling point petroleum ether, rubbing the contents of the dish with a glass rod or other suitable instrument and transferring the successive portions of solvent to a second porcelain dish. The extracted residue should finally be tested with a drop of ferric-alum solution, and if any reaction for salicylic acid be given it should be taken up in water, reextracted with ether, and the operation repeated. The gasoline extract is finally allowed to evaporate spontaneously.

Dissolve the residue in a small amount of hot water and dilute to a definite volume. Dilute aliquots of the solution and match, in Nessler tubes or with a colorimeter, the color obtained by adding a few drops of ferric-chlorid or ferric-alum solution with that of a standard solution of salicylic acid containing about 1 mg of salicylic acid in 50 cc. A 0.5 per cent solution of ferric chlorid should be used or a 2 per cent solution of ferric alum.<sup>b</sup> In either case, and especially with ferric chlorid, an excess of reagent should be avoided, although an excess of 0.5 cc of 2 per cent ferric-alum solution may be added to 50 cc of the solution of salicylic acid without impairing the results.

Salicylic acid may often be separated from fat extracted with the ether by washing the ether solution with dilute ammonium hydroxid. Then evaporate the aqueous liquid almost to dryness and test with ferric solution.

In the case of foods which yield to the gasoline solution of the ether residue a color that obscures the ferric-chlorid reaction (for example, tomatoes), the ether solution may be evaporated, the residue dried in a desiccator or in a current of dry air, sublimed, and collected on a watch glass cooled with ice. Then dissolve the sublimate in hot water and test with ferric alum.

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<sup>a</sup> In examining a substance whose ether extract does not give a color or precipitate with ferric solution, the drying of the residue and its extraction with gasoline may be omitted. The residue may then be transferred by means of warm water directly from the distilling flask to the graduated flask, in which it is made up to a definite volume. Substances interfering with the ferric reaction may often be removed by precipitation with ferric chlorid or lime, as directed on p. 181.

<sup>b</sup> This solution should be boiled until a precipitate appears, allowed to settle, and filtered. The acidity of the solution is slightly increased in this manner, but so precipitated it keeps clear for a considerable time, and the turbidity caused by its dilution with water is much less and does not appear for a much longer time than if the unboiled solution is employed. This turbidity is especially objectionable in the quantitative estimation of salicylic acid, as it interferes with the exact matching of the color.

The same difficulty may often be avoided, and in fact the extraction with gasoline of the dry residue from the ether extraction may sometimes be obviated, by precipitating before extraction with ferric chlorid or calcium chlorid, making alkaline, and filtering. By this means tannin is entirely separated from the product and other substances whose color masks the salicylic-acid reaction are often removed.

## 2. Benzoic Acid.

### (a) QUALITATIVE DETECTION.

Separate benzoic acid as directed for salicylic acid. If benzoic acid be present in considerable quantity, it will crystallize from the evaporated ether in shining leaflets with characteristic odor on heating. Dissolve the residue in hot water, divide into two portions, and test by the following methods:

#### (1) FIRST METHOD.<sup>a</sup>

Make the residue alkaline with ammonium hydroxid, expel the excess of ammonia by evaporation, take up the residue with water, and add a few drops of a neutral 0.5 per cent solution of ferric chlorid. The presence of benzoic acid will be indicated by the formation of a brownish-colored precipitate of ferric benzoate.

#### (2) SECOND METHOD <sup>a</sup> (MOHLER'S METHOD).

Evaporate to dryness and treat the residue with 2 or 3 cc of strong sulphuric acid.<sup>b</sup> Heat until white fumes appear, organic matter is charred, and benzoic acid is converted into sulpho-benzoic acid. Then add a few crystals of potassium nitrate which causes the formation of meta-dinitrobenzoic acid. When cool dilute the acid with water and add ammonium hydroxid in excess. Then cool the mixture, transfer to a test tube, and add a drop or two of fresh, colorless ammonium sulphid so that the solutions do not mix. The nitrocompound is converted into ammonium meta-diamidobenzoic acid, which possesses a red color. This reaction takes place immediately and is seen at the surface of the liquid without stirring.

### (b) QUANTITATIVE ESTIMATION.

Evaporate the ether extract obtained as directed under salicylic acid to dryness, thoroughly dry in a sulphuric acid desiccator (preferably in vacuum) and sublime under a watch glass cooled with a piece of ice or a condenser, the lower end of which is closed with a piece of rubber dam. Or the ether extract (or its solution in gasoline) may be transferred into the tube *a*, as shown in the accompanying figure, the ether or gasoline removed by a gentle current of air, the tube placed in a vacuum desiccator until its contents are thoroughly dry, and the residue sublimed at the temperature of 250° C., the sublimate being collected in tube *b*.

During the sublimation, air is drawn very slowly through the apparatus (a wash bottle is used to gauge the speed of the current) to insure the volatilized benzoic acid passing into tube *b*. The joint between the two tubes is preferably made by means of a cork stopper. The most satisfactory results are obtained by placing the tube *a* inside of an oven the temperature of which is raised grad-

<sup>a</sup> Mohler, *Bul. soc. chim.*, Paris, 1890, 3 (3): 414.

<sup>b</sup> If this is the only method employed, the sulphuric acid may be added directly to the residue left on the evaporation of the ether.

ually until it reaches  $250^{\circ}\text{C}$ . The bulb of the tube *b* should be just outside of the oven, in order that the crystals may form therein. By means of this apparatus considerably higher results are obtained than by subliming on a watch glass, as described above.

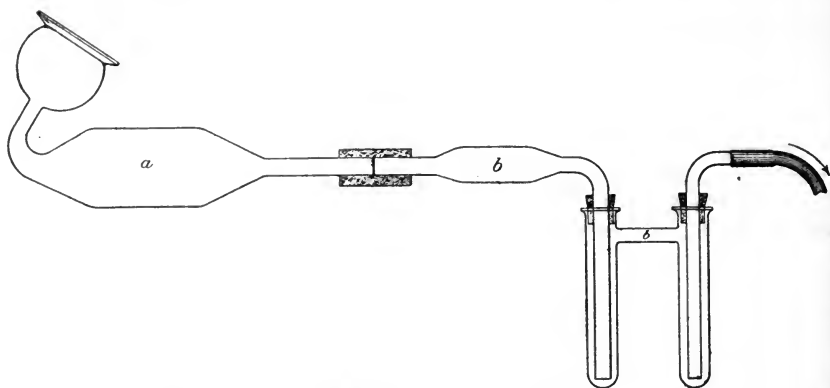


FIG. 11.—Apparatus for the determination of benzoic acid.

The sublimate of benzoic acid collected in tube *b* may be removed by solution in alcohol, and the amount confirmed by titration. A sublimate is sometimes obtained which somewhat resembles benzoic acid in appearance and which has an acid reaction. Before applying the method, therefore, to any class of foods, blank experiments should be made to determine whether a sublimate is obtained under the same conditions from the ether extract of that class of foods.

### 3. Saccharin.

#### (a) QUALITATIVE DETECTION.

Extract with ether (after maceration and exhaustion with water, if necessary), as described under salicylic acid. Allow the ether extract to evaporate spontaneously and note the taste of the residue. The presence of saccharin to the amount of 20 mg per liter is indicated by a sweet taste. This may be confirmed by heating with sodium hydroxid, as described below, and detecting the salicylic acid formed thereby. Results by this method indicating the presence of a faint trace of saccharin in wines which did not contain it have been frequently obtained, owing to the presence in wine of so-called "false saccharin."

Acidify 50 cc of a liquid food (or the aqueous extract of 50 grams of a solid or semisolid, prepared as directed on page 179) and extract with ether. Test the extracted matter in the usual way for salicylic acid, return the gasoline extract to the dish containing the residue, dilute the whole to about 10 cc volume, and add 2 cc of sulphuric acid (1:3). Bring the solution to the boiling point and add a 5 per cent solution of potassium permanganate, drop by drop, to slight excess; partly cool the solution, dissolve in it a piece of sodium hydroxid, and filter the mixture into a silver dish (silver crucible lids are well adapted to the purpose); evaporate to dryness and heat for 20 minutes at  $210^{\circ}$  to  $215^{\circ}\text{C}$ . Dissolve the residue in water, acidify and extract with ether, evaporate the ether, and test the residue with two drops of a 2 per cent solution of ferric alum. By this method all the so-called false saccharin and the salicylic acid naturally present (also added salicylic acid when not present in too large amount) are destroyed, while 5 mg of saccharin per liter is detected with certainty.

## (b) APPROXIMATE QUANTITATIVE ESTIMATION.

**Extract** as directed under the quantitative estimation of salicylic acid, page 179, but acidify with hydrochloric or phosphoric acid instead of sulphuric acid, determine the weight of sulphur in the residue, and multiply by 5.712 for the weight of saccharin, expressed in grams. The results obtained by this method are only approximate.

## 4. Boric Acid and Borates.

(a) QUALITATIVE DETECTION.<sup>a</sup>

Render decidedly alkaline with lime water about 25 grams of the sample and evaporate to dryness on a water bath. Ignite the residue to destroy organic matter. Digest with about 15 cc of water, add hydrochloric acid, drop by drop, until all is dissolved, and add 1 cc in excess. Moisten a piece of delicate turmeric paper with the solution; if borax or boric acid is present, the paper on drying will acquire a peculiar red color, which is changed by ammonium hydroxid to a dark blue-green, but is restored by acid.

A preliminary test may be made by immersing a strip of turmeric paper in about 100 cc of liquid foods, to which about 7 cc of concentrated hydrochloric acid has been added. Solid and pasty goods may be heated with enough water to make them thoroughly fluid, hydrochloric acid added in about the proportion of 1 to 13, and tested in the same manner.

(b) QUANTITATIVE ESTIMATION.<sup>b</sup>

Render 100 grams of the sample decidedly alkaline with sodium hydroxid and evaporate to dryness in a platinum dish. Ignite the residue thoroughly, heat with about 20 cc of water, and add hydrochloric acid, drop by drop, until all is dissolved. Transfer to a 100-cc flask, the volume not being allowed to exceed 50 to 60 cc. Add 0.5 gram of calcium chlorid and a few drops of phenolphthalein, then a 10 per cent solution of caustic soda until a permanent slightly pink color is produced, and finally add 25 cc of limewater. Make the volume up to 100 cc. Mix and filter through a dry filter. To 50 cc of the filtrate add normal sulphuric acid until the pink color disappears, then methyl orange, and continue the addition of the acid until the yellow is just changed to pink. Boil to expel carbon dioxid. Add fifth-normal caustic soda until the liquid assumes the yellow tinge, excess of soda being avoided. Cool the solution, add a little phenolphthalein, and an equal volume of glycerin. Titrate with standardized sodium hydroxid until a permanent pink color is produced.

One cubic centimeter of fifth-normal soda solution is equal to 0.0124 gram of crystallized boric acid.

## 5. Formaldehyde.

## (a) PREPARATION OF SAMPLE.

If the material be solid or semisolid, macerate from 200 to 300 grams in a mortar with about 100 cc of water until a sufficient degree of fluidity is obtained. Transfer to a short-necked distilling flask of copper or glass of from 500 to 800 cc capacity and make distinctly acid with phosphoric acid. Connect the flask with a glass condenser and distil from 40 to 50 cc.

<sup>a</sup> U. S. Dept. Agr., Division of Chemistry, Bul. 51, p. 113.

<sup>b</sup> Thomson's method, Sutton's Volumetric Analysis, 9th ed., p. 93.

(b) PHENYLHYDRAZIN HYDROCHLORID METHOD.<sup>a</sup>

Mix 5 cc of the distillate as prepared under (a), or of an alcoholic solution or extract from the substance under examination, with 0.03 gram of phenylhydrazin hydrochlorid, and 4 or 5 drops of a 1 per cent solution of ferric chlorid. Add slowly and with agitation, in a bath of cold water to prevent the heating of the liquid, from 1 to 2 cc of concentrated sulphuric acid. A precipitate is formed which can be dissolved by the addition of either concentrated sulphuric acid (keeping the mixture cool) or with alcohol. With meats and fats the formaldehyde should first be extracted with alcohol and the filtrate tested. In the case of fat it is necessary to heat the mixture above the melting point of the fat to insure thorough extraction. Milk is shaken with an equal volume of strong alcohol and the filtrate employed. Other liquids are shaken with an equal volume of strong alcohol and filtered in case of the formation of any insoluble matter.

In the hands of different analysts this method is found to give reliable reactions for formaldehyde in solutions of formaldehyde varying from 1 part in 50,000 to 1 part in 150,000. Acetic aldehyde and benzaldehyde give no reaction when treated by this method and do not interfere with the reaction given by formaldehyde.

(c) PHENYLHYDRAZIN HYDROCHLORID AND FERRICYANID METHOD.<sup>b</sup>

This method may be applied directly to liquid foods or to an aqueous or alcoholic extract of solid foods. To from 3 to 5 cc of liquid food or extract of the same add a lump of phenylhydrazin hydrochlorid about the size of a pea, from 2 to 4 drops (not more) of a 5 to 10 per cent solution of potassium ferrieyanid, and from 8 to 12 drops of an approximately 12 per cent solution of sodium hydroxid. The method is not applicable to preparations containing blood-coloring matter. In such cases use nitroprussid in place of the ferrieyanid. Alcoholic extracts from foods must be diluted with water to prevent the precipitation of potassium ferrieyanid.

Apply the method directly to milk without any preparation of sample. In the case of meat finely comminute the sample, extract with two volumes of hot water, and employ the liquid pressed out for the test. Warm fats above the melting point with 10 cc of alcohol (80 to 95 per cent by volume), thoroughly shake, cool, and filter through a moistened paper, using the filtrate for the examination.

When formaldehyde is present to the extent of more than 1 part in 70,000 to 80,000 in the solution tested, a distinct green or bluish green reaction is obtained. In more dilute solutions the green tint becomes less marked and a yellow tinge tending toward greenish brown is formed.

With this method acetic aldehyde and benzaldehyde give a color varying from red to brown, according to the strength of the solution. A reaction may therefore be obtained with these aldehydes similar to that obtained with formaldehyde in solutions more dilute than 1 part in 70,000. The presence of acetic aldehyde or benzaldehyde together with formaldehyde gives a yellowish or yellowish green tinge. The reaction for formaldehyde may therefore be masked by the presence of other aldehydes, but is characteristic when a clear green color is obtained.

<sup>a</sup> Arnold and Mentzel, Zts. Nahr. Genussm., 1902, 5: 353.

<sup>b</sup> Arnold and Mentzel, Chem. Ztg., 1902, 26: 246; Abs. J. Chem. Soc., 1902, 82 (2): 367; Abs. Chem. Centrbl., 1902, 73 (1): 1076.



(d) HEHNER'S METHOD.<sup>a</sup>

To the milk to be tested add strong commercial sulphuric acid without mixing, and at the junction of the two liquids a violet or blue color will appear if the milk contains one or more parts of formaldehyde per 10,000. This color is supposed to be given only when there is a trace of ferric chlorid or other oxidizing agent present. As pointed out by Hehner, milk may be treated directly by this method without any other operation, and some other articles of food rich in proteids—for example, egg albumen—give the reaction in the presence of water without the addition of milk. The distillate described above may be mixed with milk and this test applied.

## (e) LEACH'S METHOD.

Add about 5 cc of the distillate obtained under (a) to an equal volume of pure milk in a porcelain casserole and about 10 cc of concentrated hydrochloric acid, containing 1 cc of 10 per cent ferric chlorid solution, to each 500 cc of acid. Heat to 80° or 90° C. directly over the gas flame, giving the casserole a rotary motion to break up the curd. A violet coloration indicates formaldehyde.

(f) RIMINI'S METHOD.<sup>b</sup>

Treat 15 cc of milk or other liquid food under examination or of the distillate prepared as directed under (a) with 1 cc of a dilute solution of phenylhydrazin hydrochlorid, then with a few drops of dilute ferric-chlorid solution, and, finally, with concentrated hydrochloric acid. The presence of formaldehyde is indicated by the formation of a red color, which changes after some time to orange yellow.

This method is suitable for the examination of milk without previous treatment, but more delicate tests may be obtained from the distillate from milk or from milk serum. The reaction is not interfered with by acetic aldehyde or benzaldehyde.

(g) PHLOROGLUCOL METHOD.<sup>c</sup>

Prepare the reagent by dissolving 1 gram phloroglucol and 20 grams of sodium hydroxid in sufficient water to make 100 cc. To 10 cc of milk or other liquid food under examination in a test tube add, by means of a pipette, 2 cc of this reagent, placing the end of the pipette on the bottom of the tube in such a manner that the reagent will form a separate layer.

A bright red coloration (not purple) is formed at the zone of contact if formaldehyde be present. This solution gives a yellow color in the presence of some other aldehydes, and if it is used for the detection of aldehyde formed by the oxidation of methyl alcohol after the destruction of ethyl aldehyde with hydrogen peroxid an orange-yellow color will slowly appear when an insufficient amount of hydrogen peroxid has been employed. On the other hand, if the excess of hydrogen peroxid is not fully destroyed before the use of this reagent a purple color will slowly form. The clear, red color given by the use of this reagent forms quickly, and in the presence of but a small amount of formaldehyde soon fades.

<sup>a</sup> Analyst, 1895, 20: 155.

<sup>b</sup> Ann. di Farmacol., 1898, 97: Abs. Chem. Centrbl., 1898, 69 (1): 1152; Abs. J. Soc. Chem. Ind., 1898, 17: 697.

<sup>c</sup> Jorissen, Service de Surveillance des Aliments en Belgique, through Bul. soc. chim. Belg., 1897-98, 11: 12, 211; Abs. Analyst, 1897, 22: 282.

## 6. Fluorids.

### (a) MODIFIED METHOD OF BLAREZ.<sup>a</sup>

Thoroughly mix the sample and heat 150 cc to boiling (in the case of solid foods the filtrate prepared as directed under salicylic acid may be employed. Add to the boiling liquor 5 cc of a 10 per cent solution of potassium sulphate and 10 cc of a 10 per cent solution of barium acetate. Collect the precipitate in a compact mass (a centrifuge may be used advantageously) and wash upon a small filter. Transfer to a platinum crucible and ash.

Prepare a glass plate (preferably of the thin variety commonly used for lantern slide covers), as follows: First thoroughly clean, polish, and coat on one side by carefully dipping the plate while hot in a mixture of equal parts of Canauba wax and paraffin. Near the middle of the plate make a distinctive mark through the wax with a sharp instrument, such as a pointed piece of wood or ivory, which will remove the wax and expose the glass without scratching the latter.

Add a few drops of concentrated sulphuric acid to the residue in the crucible and cover with the waxed plate, having the mark nearly over the center and making sure that the crucible is firmly embedded in the wax. Place in close contact with the top or unwaxed surface of the plate a cooling device, consisting of a glass tube considerably larger in diameter than the crucible, the bottom of the tube being covered tightly with a thin sheet of pure rubber. A constant stream of cold water is passed through the tube. Heat the crucible for an hour at as high a temperature as practicable without melting the wax (an electric stove gives the most satisfactory form of heat).

Remove the glass plate and indicate the location of the distinguishing mark on the unwaxed surface of the plate by means of gummed strips of paper, then melt off the wax by heat or a jet of steam, and thoroughly clean the glass with a soft cloth. If fluorin be present, a distinct etching will be apparent on the glass where it was exposed.

### (b) SECOND METHOD.

If it is desired the preceding method may be varied by mixing a small amount of precipitated silica with the precipitated calcium fluorid and applying the method given below for the detection of fluosilicates.

This method is of value in the presence of foods whose ash contains a considerable amount of silica, which unites with fluorin and forms fluosilicates. The sulphuric acid then liberates hydrofluosillicic acid, which would escape detection by the Blarez modified method.

## 7. Fluoborates and Fluosilicates.

Make about 200 grams of the sample alkaline with lime water, evaporate to dryness, and incinerate. Extract the crude ash first obtained with water, to which sufficient acetic acid has been added to decompose carbonates, filter, burn the insoluble portion, extract with dilute acetic acid, and again filter. The insoluble portion now contains calcium silicate and fluorid, while the filtrate will contain all the boric acid present.

### (a) FIRST METHOD.<sup>b</sup>

Incinerate the filter containing the insoluble portion, mix with a little precipitated silica, and place, with the addition of 1 or 2 cc of concentrated sul-

<sup>a</sup> Chem. News, 1905, 91: 39; Ann. Rept. Mass. State Board of Health, 1905, p. 498.

<sup>b</sup> Nivière and Hubert, *Moniteur scientifique*, 1895 [4], 9: 324.

phuric acid, in a short test tube, which is attached to a small U tube containing a few drops of water. Place the test tube in a beaker of water and keep it hot on the steam bath for from 30 to 40 minutes. If any fluorid be present the silicon fluorid generated will be decomposed by the water in the U tube and will form a gelatinous deposit on the walls of the tube.

Now test the filtrate as directed under boric acid. If both hydrofluoric and boric acids be present it is probable that they are combined as borofluorid. If, however, silicon fluorid is detected and not boric acid, the operation is repeated without the introduction of the silica, in which case the formation of the silicon skeleton is conclusive evidence of the presence of fluosilicate.<sup>a</sup>

#### (b) SECOND METHOD.

Incinerate the filter containing the insoluble portion in a platinum crucible, mix with a little precipitated silica, and add 1 cc of concentrated sulphuric acid. Cover the crucible with a watch glass, to the underside of which a drop of water is suspended, and heat an hour at the temperature of 70° to 80° C.<sup>b</sup> The silicon fluorid which is formed is decomposed by the water, leaving a gelatinous deposit of silica and etching a ring at the periphery of the drop of water. Test the filtrate for boric acid as described under section 4, page 183.

### 8. Sulphurous Acid.

#### (a) QUALITATIVE DETECTION.<sup>c</sup>

To about 25 grams of the sample (with the addition of water, if necessary) placed in a 200 cc Erlenmeyer flask, add some sulphur-free zinc, and several cubic centimeters of hydrochloric acid. In the presence of sulphites hydrogen sulphid will be generated and may be tested for with lead paper. Traces of metallic sulphids are occasionally present in vegetables, and the above test will indicate sulphites. Hence positive results obtained by this method should be verified by the distillation method.

It is always advisable to make the quantitative determination of sulphites, owing to the danger that the test may be due to traces of sulphids. A trace is not to be considered sufficient indication of the presence of sulphur dioxid either as a bleaching agent or as a preservative.

#### (b) DETERMINATION OF TOTAL SULPHUROUS ACID.

##### (1) FIRST METHOD (DISTILLATION METHOD).

Distil 100 grams (adding water, if necessary) in a current of carbon dioxid after the addition of about 5 cc of a 20 per cent solution of glacial phosphoric acid until 50 cc have passed over. Collect the distillate in a tenth-normal iodine solution in a flask closed with a stopper perforated with two holes, through one of which the end of the condenser passes and through the other a U-tube containing a portion of the standardized iodine solution. Twenty-five cubic centimeters of tenth-normal iodine solution may be employed, diluted with water to give the desired volume. The method and apparatus may be simplified without material loss in accuracy by omitting the current of carbon dioxid, adding

<sup>a</sup> It must be remembered that, in an ash containing an appreciable amount of silica, sulphuric acid will liberate silicon fluorid rather than hydrofluoric acid. The presence of a fluosilicate is indicated, therefore, and not the presence of a fluorid.

<sup>b</sup> The watch glass may be kept cool by means of a piece of ice.

<sup>c</sup> U. S. Dept. Agr., Division of Chemistry, Bul. 13, pt. 8, p. 1032.

10 cc of phosphoric acid instead of 5 cc, and dropping into the distilling flask a piece of sodium bicarbonate, weighing not more than a gram, immediately before attaching to the condenser. The carbon dioxide liberated is not sufficient to expel the air entirely from the apparatus, but will prevent oxidation to a large extent. The U-tube trap may also be omitted if the end of the condenser tube is made to extend below the surface of the iodine solution, and the distillation conducted with a steady flame. When the distillation is finished wash the contents of the U-tube into the flask and determine the excess of iodine with standard thiosulphate solution. On account of its lack of permanence the iodine solution employed should be titrated from time to time with a tenth-normal thiosulphate solution (containing 24.8 grams  $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$  per liter). One cubic centimeter of tenth-normal iodine solution is equivalent to 0.0032 gram of sulphur dioxide ( $\text{SO}_2$ ).

(2) SECOND METHOD (DIRECT TITRATION METHOD).

In the examination of wine fairly accurate results may also be obtained by the following method. Care must be taken in applying the method to other products than wine to determine whether iodine is decolorized by any substance that may be naturally present.

Place 25 cc of a solution of potassium hydroxide containing 56 grams per liter in a flask of approximately 200 cc capacity. Introduce 50 cc of the sample by means of a pipette, mix with the potassium hydroxide, and allow the mixture to stand fifteen minutes with occasional agitation. Add 10 cc of 1:3 sulphuric acid and a few cubic centimeters of starch solution, and titrate the mixture with a fiftieth-normal iodine solution. Introduce the iodine solution as rapidly as possible and continue the addition until the blue color will last for several minutes. One cubic centimeter of fiftieth-normal iodine solution is equivalent to 0.00064 gram of sulphur dioxide.

(c) DETERMINATION OF FREE SULPHUROUS ACID.

(Especially adapted to wine.)

Treat 50 cc of the sample in a flask having a capacity of approximately 200 cc with about 5 cc of 1:3 sulphuric acid, add a small piece of sodium carbonate (about 0.5 gram) to expel the air, and titrate the sulphurous acid with fiftieth-normal iodine solution, as directed under total sulphurous acid.

One cubic centimeter of fiftieth-normal iodine solution is equivalent to 0.00064 gram of sulphur dioxide.

9. Beta-Naphthol.

Extract 200 cc of the sample (or of its aqueous extract prepared as directed on page 179) with 10 cc of chloroform in a separatory funnel, add a few drops of alcoholic potash to the chloroform extract in a test tube, and place in a boiling water bath for two minutes. The presence of beta-naphthol is indicated by the formation of a deep-blue color, which changes through green to yellow.

10. Abrastol.

(a) SINIBALDI'S METHOD.\*

Make 50 cc of the sample alkaline with a few drops of ammonium hydroxide and extract with 10 cc of amyl alcohol (ethyl alcohol is added if an emulsion is

\* *Moniteur scientifique*, 1893, (4), 7: 842.

formed). Decant the amyl alcohol, filter if turbid, and evaporate to dryness. Add to the residue 2 cc of a mixture of equal parts of strong nitric acid and water, heat on the water bath until half of the water is evaporated, and transfer to a test tube with the addition of 1 cc of water. Add about 0.2 cc of ferrous sulphate and an excess of ammonium hydroxid, drop by drop, with constant shaking. If the resultant precipitate is of a reddish color, dissolve it in a few drops of sulphuric acid, and add ferrous sulphate and ammonium hydroxid as before. As soon as a dark-colored or greenish precipitate has been obtained introduce 5 cc of alcohol, dissolve the precipitate in sulphuric acid, and shake the fluid well and filter. In the absence of abrastol this method gives a colorless or light-yellow liquid, while a red color is produced in the presence of 0.01 gram of abrastol.

(b) SANGLÉ-FERRIÈRE'S METHOD.<sup>a</sup>

Boil 200 cc of the sample with 8 cc of concentrated hydrochloric acid for one hour in a flask with a reflux condenser attached. Abrastol is thus converted into beta-naphthol and is detected as directed under section 9, page 188.

11. Sucrol or Dulcin.

(a) MORPURGO'S METHOD.<sup>b</sup>

Evaporate about 100 cc of the sample (or of the aqueous extract prepared as directed on page 179) to a sirupy consistency after the addition of about 5 grams of lead carbonate, and extract the residue several times with alcohol of about 90 per cent; evaporate the alcohol extract to dryness; extract the residue with ether, and allow the ether to evaporate spontaneously in a porcelain dish. Add 2 or 3 drops each of phenol and concentrated sulphuric acid and heat for about 5 minutes on the water bath; cool; transfer to a test tube and pour ammonium hydroxid or sodium hydroxid over the surface with the least possible mixing. The presence of dulcin is indicated by formation of a blue zone at the plane of contact.

(b) JORISSEN'S METHOD.<sup>c</sup>

Suspend the residue from the ether extract obtained as directed above in about 5 cc of water; add from 2 to 4 cc of an approximately 10 per cent solution of mercuric nitrate, and heat from 5 to 10 minutes on the water bath. In the presence of sucrol a violet-blue color is formed, which is changed to a deep violet by the addition of lead peroxid.

<sup>a</sup> Comp. rend., 1893, 117: 796.

<sup>b</sup> Zts. anal. Chem., 1896, 35: 104.

<sup>c</sup> Ibid., p. 628.

## XXVIII. METHODS FOR THE DETECTION AND DETERMINATION OF COLORING MATTER.—PROVISIONAL.

### 1. General Discussion.

In the manufacture of coal-tar dyes many become contaminated with poisonous metals, such as arsenic, copper, zinc, tin, and lead. There is always the possibility of the presence of arsenic, as sulphuric acid is used at one stage or another in the preparation of nearly every dye.

Some colors have metallic atoms in their molecule, such as malachite green, which is a double chlorid of zinc in combination with the organic group.

Many vegetable colors are sold as lakes of tin or alum. Other colors are known to have a toxic action, such as picric acid and naphthol yellow.

### 2. Detection of Artificial Colors by Dyeing Wool.<sup>a</sup>

#### (a) METHOD OF SOSTEGNI AND CARPENTIERI.<sup>b</sup>

Dissolve from 10 to 20 grams of the sample in 100 cc of water, filter if necessary, acidify with from 2 to 4 cc of a 10 per cent solution of hydrochloric acid. In this solution immerse a piece of woollen cloth, which has been washed in a very dilute solution of boiling potassium hydroxid and then washed in water, and boil for from 5 to 10 minutes. Remove the cloth, thoroughly wash it in water, and boil in a very dilute hydrochloric acid solution. After washing out the acid dissolve the color in a solution of ammonium hydroxid (1:50). With some of the dyes solution takes place quite readily while with others it is necessary to boil for some time. Take the wool out, add a slight excess of hydrochloric acid to the solution, immerse another piece of wool, and boil it again.

With vegetable coloring matter this second dyeing gives practically no color, and there is no danger of mistaking a fruit color for one of coal-tar origin. It is absolutely necessary that the second dyeing should be made, as some of the coal-tar dyes<sup>c</sup> will dye a dirty orange in the first acid bath which might be easily passed for vegetable color, but after solution in alkaline bath the second acid bath dyes a bright pink.\*

<sup>a</sup> The method of Sostegni and Carpentieri and Arata's method for coal-tar colors are not reliable in the presence of archil, archil derivatives, and sulphonated indigo, as these substances give dyeing reactions not to be distinguished from coal-tar colors. It is, however, comparatively easy to detect archil. A red color turning purple with dilute ammonium hydroxid, reduced by zinc and hydrochloric acid and easily reoxidizing in the air, is either archil or a closely related color. (Tolman, J. Amer. Chem. Soc., 1905, 27: 25.) Archil can be extracted from an ammoniacal solution by amyl alcohol, but the sulphonated archil colors now on the market do not respond to the test. Indigo is used in many green and violet colors and can be recognized as described by Allen. (Com. Org. Anal., 3: (1) 525-541.

<sup>b</sup> Zts. anal. Chem., 1896, 35: 397.

<sup>c</sup> U. S. Dept. Agr., Bureau of Chemistry, Bul. 66, p. 24.

(b) ARATA'S METHOD.<sup>a</sup>

This method gives results comparable with those of the first dyeing of the preceding method. It was recommended for detecting coal-tar colors in wine, and has been used by Winton<sup>b</sup> in fruit products.

Boil from 20 to 30 grams of the sample dissolved in 100 cc of water for ten minutes with 10 cc of a 10 per cent solution of potassium bisulphate and a piece of white wool or woollen cloth, which has been previously heated to boiling in a very dilute solution of sodium hydroxid and thoroughly washed in water. After removal from the solution, wash the wool in boiling water and dry it between filter papers. If the coloring matters are entirely from the fruit, the wool will be either uncolored or will take on a faint pink or brown which is changed to green or yellow by ammonium hydroxid and not restored by washing.

In addition to this, it is advisable in all cases to dissolve out the coloring matter with ammonium hydroxid as in the first method and dye again, since Arata's method gives practically the same results as the first dyeing in hydrochloric-acid bath and needs to be confirmed by the second dyeing.

Another advantage in the second dyeing is that if a large piece of woollen cloth is used in the first dyeing, and a small piece in the second dyeing, small amounts of coloring matter can be brought out much more decidedly in the second dyeing, where practically all of the vegetable coloring matter has been excluded. The coloring matter can be identified to a certain extent by the schemes of Witt,<sup>c</sup> Allen,<sup>d</sup> Weingärtner,<sup>e</sup> Dommergue,<sup>f</sup> Girard and Dupré,<sup>g</sup> and Rota.<sup>h</sup> The tests can be made directly on the dyed fabric or the dye can be dissolved out.<sup>i</sup> To remove the color, wash the wool with dilute tartaric acid and then with water and dry between filter paper. Saturate the wool with strong sulphuric acid, press out the color with a glass rod after from five to ten minutes, and dilute to 10 cc with water.

Remove the wool, make solution alkaline with ammonium hydroxid, and when cold extract with from 5 to 10 cc of amyl alcohol. Separate the amyl alcohol, evaporate it to dryness, and test the residue with strong sulphuric acid.

Ponceau R, 2R, 3R, S, and 3S give yellow red to carmine red.

Ponceau S and tropæolin O give yellow to orange yellow.

Biebrich scarlet gives a green; Bordeaux red and crocein scarlet give blue; tropæolin OOO and solid red give violet.

If the wool is well dyed, most of these colors may be obtained on the fabric.

These are the reactions of only a few of the more common colors; in order to carry the work further the more complete works mentioned must be used.

<sup>a</sup> Zts. anal. Chem., 1889, 28: 639.

<sup>b</sup> Conn. Agr. Exper. Stat. Rept., 1899, Part 2, p. 131.

<sup>c</sup> Zts. anal. Chem., 1887, 26: 100.

<sup>d</sup> Commercial Organic Analysis, 3 (1): 399-420.

<sup>e</sup> Zts. anal. Chem., 1888, 27: 232-249.

<sup>f</sup> Zts. anal. Chem., 1890, 29: 369-377.

<sup>g</sup> Analyse des matières alimentaires, etc., 583-593.

<sup>h</sup> Analyst, 1899, 24: 41.

<sup>i</sup> Zts. anal. Chem., 1889, 28: 639; Borgmann, Analyse des Weines, p. 91; Winton, Conn. Agr. Exper. Stat. Rept., 1899, Part 2, p. 131.

### 3. Detection of Coal-Tar Colors by Extraction with Solvents.<sup>a</sup>

In the Paris Municipal Laboratory <sup>b</sup> the following scheme of extraction of coal-tar colors is used.

The acid colors, sulphu-fuchsin, azo derivatives, and phthaleins are not precipitated by tannin and are insoluble or only slightly soluble in acetic ether or amyl-alcohol.

The basic colors (fuchsin, safranin, etc.), are precipitated by tannin and readily soluble in acetic ether or amyl-alcohol.

(a) To 50 cc of wine add ammonium hydroxid in slight excess; then add 15 cc of amyl-alcohol, shake, and allow to stand.

(1) If the alcohol is colored red or violet, decant, wash, filter, evaporate to dryness in presence of a piece of wool, and test the dyed wool with sulphuric acid.

(2) If the alcohol is not colored, separate, and add acetic acid. If the alcohol becomes colored the presence of basic anilin color is indicated.

(3) If the amyl-alcohol is uncolored, both before and after the addition of acetic acid, no basic coal-tar color is present.

(b) Add an excess of calcined magnesia and then a 20 per cent solution of mercuric acetate and bring to a boil. A coloration before or after addition of acetic acid indicates the presence of coal-tar dyes, particularly acid dyes.

(c) Extract the solution with acetic ether made alkaline by barium hydroxid. This dissolves basic colors.

In any case the colors must be fixed on wool, as many of the fruit colors are extracted and will give reactions with sulphuric acid, which may be mistaken for coal-tar colors.

The extraction of fruit colors is shown in the following tables, the first of which was prepared by Truchon and Martin-Claude,<sup>c</sup> and the second by Tolman. The fresh fruit juice was very slightly acidified by hydrochloric acid before extraction. In no case in the dyeing test was there any danger of mistaking the vegetable color for one of coal-tar origin where the double-dyeing method was used.

#### *Extraction of fruit colors with amyl-alcohol.*

Fruit.	Coloration of acid solution. <sup>d</sup>		Coloration of ammoniacal solution.		Addition of a drop of H <sub>2</sub> SO <sub>4</sub> to dyed fabric.
	Juice.	Amyl-alcohol extract.	Juice.	Amyl-alcohol extract.	
Early cherries.....	Red .....	Yellow .....	Green .....	Uncolored....	Yellow.
Ripe cherries.....	Red .....	Uncolored....	Green .....	Uncolored....	Yellow.
Early strawberries ..	Red .....	Rose .....	Green .....	Uncolored....	Rose.
Ripe strawberries ..	Red .....	Red .....	Green .....	Uncolored....	Rose (dyes silk a rose red).
Raspberries .....	Red .....	Red .....	Green .....	Uncolored....	Dyes silk rose. Uncolored.
Red currants .....	Red .....	Uncolored....	Green .....	Uncolored....	
White currants .....	White .....	Uncolored....	Brown .....	Uncolored....	
Black currants .....	Dark red ..	Red .....	Deep green ..	Uncolored....	
Peaches .....	Yellow ..	Uncolored....	Brown .....	Yellow-red...	
Pears .....	Yellow ..	Uncolored....	Brown .....	Yellow-red...	
Quinces.....	Yellow ..	Uncolored....	Brown .....	Yellow-red...	
Apples .....	Yellow ..	Uncolored....	Brown .....	Yellow-red...	
Apricots.....	Yellow ..	Uncolored....	Brown .....	Yellow-red...	
Greengage plums....	Yellow ..	Uncolored....	Brown .....	Yellow-red...	

<sup>a</sup> See also the following circulars of the Bureau of Chemistry: No. 25, Coloring Matters for Foodstuffs and Methods for Their Detection, by W. G. Berry; No. 35, Report on Colors: The Solubility and Extraction of Colors and the Color Reactions of Dyed Fiber and of Aqueous and Sulphuric-Acid Solutions, by H. M. Loomis.

<sup>b</sup> Girard and Dupré, *Analyse des matières*, etc., p. 167.

<sup>c</sup> J. pharm. chim., 1901, 13: 174.

<sup>d</sup> Acidity of the juice.



*Extraction of fruit colors with amyl-alcohol and with ether.*

Fruit.	Color with $\text{NH}_4\text{OH}$ .	Ether extract from acid solution.	Amyl-alcohol extract from acid solution.	Dyeing tests on the juice.
Strawberry .....	Purple.....	None .....	Deep red .....	Color washed out.
Red raspberry.....	Purple.....	None .....	Deep red .....	All color does not wash out, but does not dye in the second acid bath.
Blackberry .....	Blue-purple ..	None .....	Very deep red.	Dyes purplish red in acid solution, but does not dye in the second acid bath.
Cherry .....	Purple.....	None .....	Red .....	Do.
Blackberry .....	Blue-purple ..	None .....	Red .....	Do.
Wild dewberry .....	Blue-purple ..	None .....	Red .....	Do.
Currant.....	Blue-purple ..	None .....	Red .....	Do.

It will be seen from these two tables that amyl-alcohol, as a rule, extracts fruit-coloring matter from acid solution, while ether does not. Neither amyl-alcohol nor ether extracted any color from alkaline solution of the fruit juice.

**4. Detection of Acid Magenta—Girard's Method.<sup>a</sup>**

Add to 100 cc of solution to be tested 2 cc of potassium hydroxid (5 to 100). If this does not neutralize the acid, add enough to do it. Then add 4 cc of mercuric acetate (10 to 100), agitate, and filter. The filtrate should be colorless and slightly alkaline. Acidify with a slight excess of dilute sulphuric acid, and if the solution remains uncolored there is no acid magenta present. If it becomes a light violet-red and there has been no other dye shown by the amyl-alcohol extracts, the presence of acid magenta is shown.

Acid magenta in acid solution dyes wool a magenta red. Wool dyed with it is turned yellow by strong hydrochloric acid, decolorized by ammonium hydroxid and regains its color when washed with water.

**5. Test for Martius Yellow or Naphthalene Yellow.**

Extract with 95 per cent alcohol from an acidulated sample. Evaporate the alcoholic solution to dryness with a piece of wool, which will be dyed a bright yellow, and test the dyed wool. Both sulphuric and hydrochloric acids completely decolorize it.

**6. Rota's Method of Identification of Organic Coloring Matter.<sup>b</sup>**

The coloring matters are divided into four groups by the use of stannous chlorid and hydrochloric acid and of caustic potash.

The reagents are a 10 per cent solution of stannous chlorid and a 20 per cent solution of caustic potash.

Dilute the aqueous or alcoholic solution of coloring matter to about 1 to 10,000. This strength is not of vital importance, but the color must not be too deep, as it will mask the reduction in some cases, as with safranins, where it is slow and not complete. Add to the solution a few drops of stannous chlorid

<sup>a</sup> Girard and Dupré, *Analyse des matières alimentaires*, etc., p. 169; Winton, *Conn. Agr. Exper. Stat. Rept.*, 1899, part 2, p. 132.

<sup>b</sup> Chem. Ztg., 1898, 22: 437-442; Analyst, 1899, 24: 41.

and a few drops of hydrochloric acid, shake, and heat to boiling. Care must be taken to carry along for comparison a solution of the coloring matter acidified with hydrochloric acid, in order not to mistake the action of the acid alone for reduction. Some of the colors—for instance, safranin and indulins—are slow to be reduced and must be allowed to stand for some time. For the stannous chlorid and hydrochloric acid can be substituted a solution of tin in strong hydrochloric acid.

As soon as the group is determined it is possible to carry the work further by reference to tables of coloring matter<sup>a</sup> in which the physical, chemical, and tinctorial properties are given; but it is impossible for the published books to keep up with the new dyes which are constantly being discovered, so that the tables are never complete, although they will, as a rule, contain all the data necessary.

*Classification of organic coloring matters.*

[A portion of the aqueous or alcoholic solution is treated with HCl and SnCl<sub>2</sub>.]

Complete decolorization. Reducible coloring matters. Colorless solution is treated with Fe <sub>2</sub> Cl <sub>6</sub> or shaken, with exposure to air.		The color changed no further than with HCl alone. Non-reducible colors. A part of original solution is mixed with 20 per cent KOH and warmed.	
The liquid remains unchanged. Not reoxidizable coloring matters.	The original color restored. Reoxidizable coloring matters.	Decolorization or a precipitate. Imido-carbo-quinone coloring matters.	No precipitation. Liquid becomes more colored. Oxy-carbo-quinone coloring matters.
CLASS I.	CLASS II.	CLASS III.	CLASS IV.
Nitro, nitroso, and azo colors, including azoxy and hydrazo colors. Pieric acid, naphthol yellow, Ponceau, Bordeaux, and Congo red.	Indogenide and imido-quinone coloring matters, methylene blue, safranin, indigo-carmin.	Amido-derivatives of di and triphenyl-methane, auramines, acridines, quinolines, and color derivatives of thio benzenil. Fuchsin, rosaniline, auramine.	Nonamide diphenyl-methane, oxy-ketone, and most of natural organic coloring matters. Eosines, aurin, alizarin.

<sup>a</sup> Schultz and Julius, Tabellarische Übersicht der künstlichen organischen Farbstoffe; Allen, Commercial Organic Analysis, 3d ed., 3 (1): 529-565.

*Characteristics of organic coloring matters.*

CLASS I.—REDUCED BY  $\text{HCl} + \text{SnCl}_2$  AND NOT REOXIDIZABLE.

*Nitro-coloring matters.*



Yellow or orange, soluble in water. Wool and silk dyed directly, but not cotton. The aqueous solution shows tendency to decolorization with  $\text{HCl}$ . With  $\text{HCl} + \text{SnCl}_2$  partially reduced, giving red nitro-amido derivatives (nitramines) or nitro-phenols turning red in  $\text{KOH}$ .

*Nitro-coloring matters.*



Brown or green, usually insoluble in water; indirect for fibers; with  $\text{H}_2\text{SO}_4 + \text{CHOH}$  give blue color (Liebermann's reactions).

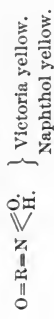
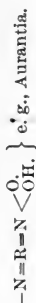
*Azo-coloring matters.*



Their aqueous solution decomposed with  $\text{KOH}$  and extracted with ether gives an ethereal extract with annexed characteristics.

Nitramines; soluble in ether in the presence of  $\text{KOH}$ .

Nitro-phenols; insoluble in ether in the presence of  $\text{KOH}$ .  
 Nonsulphonated; soluble in ether in presence of acetic acid.  
 Sulphonated; insoluble in ether.



Nonsulphonated; insoluble in water; soluble in alcohol; soluble in ether in presence of acetic acid.	Nonsulphonated amido-azo coloring matters.	$-\text{N}=\text{R}=\text{N}-\text{NHR}$ .	Dioxine (L). Naphthol green.
Sulphonated; soluble in water; insoluble in ether.	Oxyazo coloring matter without carboxyl.	$\text{O}=\text{R}=\text{N}-\text{HR}_1$ .	Bismarck brown.
Colored; shaken with dilute acetic acid yields to it the original color. Basic coloring matters.	Oxyazo coloring matters with carboxyl group.	Indirect for cotton wool.	Sudan 1 (A).
Colored solution; not yielding its color to dilute acetic acid. Neutral coloring matter.	Nonsulphonated; extracted by ether from dilute solution in acetic acid.	Direct for cotton wool.	Diamond yellow (By).
Colorless solution; yields nothing to acetic acid. A cold coloring matter.	Sulphonated; not extracted by ether from solution in dilute acetic acid.	Indirect for cotton wool.	Chrysamine.
	Nonamido compounds unaltered by $\text{HNO}_3$ .	Direct for cotton wool.	Bordeaux B (A).
	Amido compound; changed by $\text{HNO}_3$ .	Indirect for cotton wool.	Azo blue (A).
		Direct for cotton wool.	Solid yellow N (P).
			Congo red (A).

*Characteristics of organic coloring matters—Continued.*

CLASS II.—REDUCED BY HCl + SnCl<sub>2</sub> AND REOXIDIZABLE.

The ethereal solution is colored or colorless, and yields the original color to 5 per cent acetic acid. Basic coloring matters fixed on wool in alkaline bath.	The solution is readily reduced by HCl+SnCl <sub>2</sub> in the cold.	Oxyazines (no sulphur).		Nile blue A (B).
The colored solution is reduced but slowly and incompletely even on warming and with the addition of much SnCl <sub>2</sub> +HCl.		Thiazines (sulphur).		Methylene blue.
Colored; does not yield the color to acetic acid. Neutral coloring matters. Insoluble in water. Soluble in alcohol. Fixed on fibers in bath.	Blue coloring matters changed by HCl on warming.	Indulines; blue color with conc. H <sub>2</sub> SO <sub>4</sub> . Blue on dilution.		Induline soluble in alcohol.
Uncolored; yields nothing to acetic acid. Acid coloring matters. Soluble in water. Fixed on wool in acid bath.	Red or blue coloring matters. Unaltered by HCl. With HNO <sub>3</sub> yield isatin.	Safranins; green color with H <sub>2</sub> SO <sub>4</sub> . On dilution blue; then violet.		Safranin T, extra (A).
Nonsulphonated. Soluble in ether in presence of acetic acid.	Blue coloring matters changed by HCl on warming.	Indophenols.		Indophenol.
The ethereal solution of alcoholic solution is treated with KOH and extracted with ether.	Reduced by SnCl <sub>2</sub> +HCl.	Indogenides.		Indigotin.
The aqueous or alcoholic solution washed with water has the annexed characteristics.	Not reduced by SnCl <sub>2</sub> +HCl.	Oxazones.		Fluorescent blue; oreocin.

*Characteristics of organic coloring matters—Continued.*

CLASS III.—COLORING MATTERS NOT REDUCED BY  $\text{SnCl}_2 + \text{HCl}$ . CONTAINING THE IMIDO-QUINONE CARBON CHROMOPHORE— $\text{N}=\text{R}=\text{C}=\text{O}$ .

<p>The ethereal solution is colorless or colored. The color is yielded to 5 per cent acetic acid.</p> <p>Basic coloring matters. Fixed on wool in alkaline bath (<math>\text{NH}_3</math>).</p>	<p>The colorless ethereal solution does not yield its color to acetic acid.</p> <p>Neutral coloring matters. Insoluble in water. Soluble in alcohol.</p>	<p>The ethereal solution is colorless. Yellow color yielded to acetic acid nonfluorescent. The aqueous solution is decolorized by <math>\text{KOH}</math> and decomposed by <math>\text{HCl}</math>.</p>	<p>Auramines.</p>	<p>E. g., Auramine O (B).</p>
<p>The ethereal solution is colorless or colored. The color is yielded to 5 per cent acetic acid.</p> <p>Basic coloring matters. Fixed on wool in alkaline bath (<math>\text{NH}_3</math>).</p>	<p>The colorless ethereal solution does not yield its color to acetic acid.</p> <p>Neutral coloring matters. Insoluble in water. Soluble in alcohol.</p>	<p>Colorless ethereal solution. Green fluorescence. Aqueous solution precipitated by <math>\text{KOH}</math> hardly altered by <math>\text{HCl}</math>. Turns red with <math>\text{HNO}_3</math>.</p>	<p>Acridines.</p>	<p>Phosphine.</p>
<p>The ethereal solution is colorless or colored. The color is yielded to 5 per cent acetic acid.</p> <p>Basic coloring matters. Fixed on wool in alkaline bath (<math>\text{NH}_3</math>).</p>	<p>The colorless ethereal solution does not yield its color to acetic acid.</p> <p>Neutral coloring matters. Insoluble in water. Soluble in alcohol.</p>	<p>Colorless or colored ethereal solution. Nonfluorescent. Color yielded to acetic acid—reddish violet, blue, and green—without fluorescence. Aqueous solution usually decolorized on warming with <math>\text{KOH}</math>, and colored yellow by <math>\text{HCl}</math> (excepting fuchsin).</p>	<p>Fuchsines (nonsulphonated).</p>	<p>Fuchsin.</p>
<p>The ethereal solution is colorless or colored. The color is yielded to 5 per cent acetic acid.</p> <p>Basic coloring matters. Fixed on wool in alkaline bath (<math>\text{NH}_3</math>).</p>	<p>The colorless ethereal solution does not yield its color to acetic acid.</p> <p>Neutral coloring matters. Insoluble in water. Soluble in alcohol.</p>	<p>Ethereal solution colorless and nonfluorescent. Acetic acid colored rose and fluoresces.</p>	<p>Pyronines (colored yellow by <math>\text{HCl}</math>. Direct for cotton wool).</p>	<p>Pyronine (G).</p>
<p>The ethereal solution is colorless or colored. The color is yielded to 5 per cent acetic acid.</p> <p>Basic coloring matters. Fixed on wool in alkaline bath (<math>\text{NH}_3</math>).</p>	<p>The colorless ethereal solution does not yield its color to acetic acid.</p> <p>Neutral coloring matters. Insoluble in water. Soluble in alcohol.</p>	<p>Aqueous solution decolorized with <math>\text{KOH}</math>.</p>	<p>Rhodamines (nonsulphonated. Unaltered by <math>\text{HCl}</math>).</p>	<p>Rhodamine S (Br).</p>
<p>The ethereal solution is colorless or colored. The color is yielded to 5 per cent acetic acid.</p> <p>Basic coloring matters. Fixed on wool in alkaline bath (<math>\text{NH}_3</math>).</p>	<p>The colorless ethereal solution does not yield its color to acetic acid.</p> <p>Neutral coloring matters. Insoluble in water. Soluble in alcohol.</p>	<p>The ethereal solution is yellow and nonfluorescent. Alcoholic solution yellow, nonfluorescent, and unaltered by aqueous acids and alkalis.</p>	<p>Quinone-phthalones.</p>	<p>Quinoline yellow (soluble in alcohol).</p>
<p>The ethereal solution is colorless or colored. The color is yielded to 5 per cent acetic acid.</p> <p>Basic coloring matters. Fixed on wool in alkaline bath (<math>\text{NH}_3</math>).</p>	<p>The colorless ethereal solution does not yield its color to acetic acid.</p> <p>Neutral coloring matters. Insoluble in water. Soluble in alcohol.</p>	<p>Yellow coloring matters. No fluorescence in water. Unaltered by aqueous acids and alkalis.</p>	<p>Sulphonated quinone-phthalones.</p>	<p>Quinoline yellow (soluble in water).</p>
<p>The ethereal solution is colorless or colored. The color is yielded to 5 per cent acetic acid.</p> <p>Basic coloring matters. Fixed on wool in alkaline bath (<math>\text{NH}_3</math>).</p>	<p>The colorless ethereal solution does not yield its color to acetic acid.</p> <p>Neutral coloring matters. Insoluble in water. Soluble in alcohol.</p>	<p>Reddish violet, blue, or green coloring matters. Usually decolorized by <math>\text{KOH}</math>, little changed by <math>\text{HCl}</math>.</p>	<p>Sulphonated fuchsines.</p>	<p>Fuchsin S (B).</p>
<p>The ethereal solution is colorless or colored. The color is yielded to 5 per cent acetic acid.</p> <p>Basic coloring matters. Fixed on wool in alkaline bath (<math>\text{NH}_3</math>).</p>	<p>The colorless ethereal solution does not yield its color to acetic acid.</p> <p>Neutral coloring matters. Insoluble in water. Soluble in alcohol.</p>	<p>Red or violet coloring matters. Soluble in water with fluorescence. Precipitated by <math>\text{HCl}</math>. Changed but little, or not at all, by <math>\text{KOH}</math>.</p>	<p>Sulphonated rhodamines.</p>	<p>Violamine R (M).</p>
<p>The ethereal solution is colorless or colored. The color is yielded to 5 per cent acetic acid.</p> <p>Basic coloring matters. Fixed on wool in alkaline bath (<math>\text{NH}_3</math>).</p>	<p>The colorless ethereal solution does not yield its color to acetic acid.</p> <p>Neutral coloring matters. Insoluble in water. Soluble in alcohol.</p>	<p>Brownish yellow or orange coloring matters. Aqueous solution <math>\pm</math> fluorescent. Fixed directly on silk, wool, and cotton.</p>	<p>Thiazoles.</p>	<p>Primulin (B).</p>

The aqueous or alcoholic solution treated with  $\text{KOH}$  and extracted with ether.



### 7. Determination of Vegetable Colors.

A great many tests for vegetable colors are given, depending largely on color reactions with different reagents, but these must be used with very great discrimination, as they depend very largely on a fine judgment of shades of colors which many eyes are not able to distinguish.

A great deal of work has been done on detection of vegetable colors,<sup>a</sup> but in only a very few cases are the reactions specific enough to be decisive.

### 8. Detection of Turmeric.<sup>b</sup>

Extract the color with alcohol. Dip a piece of filter paper into this tincture and dry at 100° C.

Then moisten in a weak solution of boric acid to which a few drops of hydrochloric acid have been added. On drying this a cherry-red color will be developed in the presence of turmeric, which is characteristic.

### 9. Detection of Caramel.

#### (a) AMTHOR TEST.<sup>c</sup>

Place 10 cc of the solution to be tested in a high narrow glass with perpendicular sides, as, for example, a small bottle; add from 30 to 50 cc of paraldehyde, depending on the intensity of the coloring, and enough absolute alcohol to make the solutions mix. In the presence of caramel a brownish-yellow to dark-brown precipitate will collect in the bottom of the glass. Decant the liquor, wash once with absolute alcohol, dissolve in a small amount of hot water, and filter. The color of this solution will give some idea of the amount of caramel present.

It is not allowable to concentrate the solution by evaporation on a steam bath, as caramel may be formed; if it is necessary to concentrate, it must be done over sulphuric acid or at diminished pressure.

In order to identify the color, pour the solution into a freshly prepared solution of phenylhydrazin (2 parts phenylhydrazin-hydrochloric, 3 parts sodium acetate, and 20 parts of water). The presence of a considerable quantity of caramel gives a dark-brown precipitate in the cold, which is hastened by heating a little. In the case of a very small amount it takes some hours for it to collect.

#### (b) CRAMPTON AND SIMONS' FULLER'S EARTH METHOD.<sup>d</sup>

Add 25 grams of fuller's earth to 50 cc of the sample under examination, beat the mixture in a beaker, and let it stand covered half an hour at room temperature, then filter. The determination of the figure representing the color is made with the tintometer upon the liquid before and after treatment and the difference between the two results gives the percentage of color absorbed.

<sup>a</sup> Girard and Dupré, *Analyse des matières alimentaires*, etc., 580-581, also 169; A. W. Blythe, *Foods, their Composition and Analysis*, p. 91-109; Allen, *Commercial Organic Analysis*, 3 (1): p. 376. Burcker, *Traité des falsifications et alterations des substances alimentaires, et des boissons*, p. 160; W. Lenz, *Zts. anal. Chem.*, 1885, 24: 285.

<sup>b</sup> Allen, *Commercial Organic Analysis*, 3 (1): p. 359; U. S. Dept. Agr., *Division of Chemistry*, Bul. 51, p. 131.

<sup>c</sup> *Zts. anal. Chem.*, 1885, 24: 30; Borgmann, *Analyse des Weines*, p. 98.

<sup>d</sup> *J. Amer. Chem. Soc.*, 1899, 21: 355.

## 10. Detection of Cochineal.

Cochineal is used to a certain extent as a coloring matter in foods, and a very satisfactory test for it is that given in Girard and Dupré.<sup>a</sup> Dissolve the food product in water, filtering if necessary. Acidulate with hydrochloric acid and extract—with amyl alcohol, which is colored yellow or orange, the depth of color depending on the quantity of cochineal present. Separate the amyl alcohol and wash until neutral. Then separate into two portions; to the first add drop by drop a very dilute solution of uranium acetate, shaking thoroughly after each addition. In the presence of cochineal a characteristic emerald-green color is produced.<sup>b</sup>

To the second portion add a drop or so of ammonium hydroxid, and in presence of cochineal a violet coloration results. This, however, is not so sensitive to very small amounts as the first tests, and many fruit colors give tests hardly to be distinguished.

Cochineal carmine is liable to contain tin, as it is often a tin lake, although alum is also used. It is also liable to adulteration with lead compounds.

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<sup>a</sup>Analyse des matières alimentaires, etc., p. 580.

<sup>b</sup>This reaction has been tested on a number of amyl alcohol extracts from fruits, and in no case was there any chance of mistake in the reaction. Most fruits give a brown color, while blackberries and currants give a bluish color.



## XXIX. METHODS FOR THE ANALYSIS OF DRUGS.—PROVISIONAL.

[See Appendix, p. 258, for methods for alkaloids.]

### 1. Assay of Opium.<sup>a</sup>

#### (a) REAGENTS.

- (1) *Ammonia water*, 3.5 cc.
- (2) *Alcohol, ether, distilled water, and lime water* in sufficient quantities.

#### (b) DETERMINATION.

Introduce 10 grams of opium (which if fresh, should be in very small pieces, and if dry, in very fine powder) into an Erlenmeyer flask having a capacity of about 300 cc, add 100 cc of distilled water, stopper the flask and agitate it every ten minutes (or continuously in a mechanical shaker) during three hours. Then pour the contents as evenly as possible upon a wetted filter having a diameter of 12 cm, and, when the liquid has drained off, wash the residue with distilled water, carefully dropped upon the edges of the filter and its contents, until 150 cc of filtrate have been obtained. Then carefully transfer the moist opium back to the flask by means of a spatula, add 50 cc of distilled water, agitate it thoroughly and repeatedly during fifteen minutes, and return the whole to the filter. When the liquid has been drained off, wash the residue as before, until the second filtrate measures 150 cc, and finally collect about 20 cc more of a third filtrate. Evaporate carefully in a tared dish, first, the second filtrate to a small volume, then add the first filtrate, rinsing the vessels with the third filtrate, and continue the evaporation until the residue weighs 14 grams. Rotate the concentrated solution about in the dish until the rings of extract are redissolved, pour the liquid into a tared Erlenmeyer flask, having a capacity of about 100 cc, and rinse the dish with a few drops of water at a time until the entire solution, after the rinsings have been added to the flask, weighs 20 grams. Then add 10 grams (or 12.2 cc) of alcohol, shake the flask well, add 25 cc of ether, and repeat the shaking. Now add the ammonia water from a graduated pipette or burette, stopper the flask with a sound cork, shake it thoroughly during ten minutes, and then set it aside, in a moderately cool place, for at least six hours, or overnight.

Remove the stopper carefully, and should any crystals adhere to it, brush them into the flask. Place in a small funnel two rapidly acting filters, of a diameter of 7 cm, plainly folded, one within the other (the triple fold of the inner filter being laid against the single side of the outer filter), wet them well with ether, and decant the ethereal solution as completely as possible upon the inner filter. Add 10 cc of ether to the contents of the flask, rotate it, and again decant the ethereal layer upon the inner filter. Repeat this operation with another portion of 10 cc of ether. Then pour the liquid in the flask into the filter, in portions, in such a way as to transfer the greater portion of the crystals to the filter, and when the liquid has passed through, transfer the remaining crystals to the filter by washing the flask

<sup>a</sup> U. S. Pharmacopœia, 8 Rev., p. 329.

with several portions of water, using not more than 15 cc in all. Use a feather or rubber-tipped glass rod to remove the crystals that adhere to the flask. Allow the double filter to drain, then apply water to the crystals, drop by drop, until they are practically free from mother-liquor, and afterwards wash them, drop by drop, from a pipette, with alcohol previously saturated with powdered morphine. When this has passed through, displace the remaining alcohol by ether, using about 10 cc or more, if necessary. Allow the filter to dry in a moderately warm place, at a temperature not exceeding 60° C. (140° F.) until its weight remains constant, then carefully transfer the crystals to a tared watch-glass and weigh them.

Place the crystals (which are not quite pure) in an Erlenmeyer flask, add lime water (10 cc for each 0.1 gram of morphine) and shake flask at intervals during half an hour. Pass the liquid through two counterpoised rapidly acting, plainly folded filters, one within the other (the triple fold of the inner filter being laid against the single fold of the outer filter), rinse the flask with more lime water and pass the washings through the filter until the filtrate, after acidulating, no longer yields a precipitate with mercuric potassium iodid T. S. Press the filters until nearly dry between bibulous paper and dry them to a constant weight, then weigh the contents, using the outer filter as a counterpoise. Deduct the weight of the insoluble matter on the filter from the weight of the impure morphine previously found. The difference, multiplied by 10, represents the percentage of crystallized morphine contained in the opium.



# XXX. REFERENCE TABLES.

TABLE I.—*Specific gravity and percentage of alcohol.*

[According to Squibb.]

Per cent alcohol by volume.	Specific gravity.		Per cent alcohol by volume.	Specific gravity.		Per cent alcohol by volume.	Specific gravity.	
	At 15.56° C.	At 25° C.		At 15.56° C.	At 25° C.		At 15.56° C.	At 25° C.
1	0.9985	0.9970	36	0.9578	0.9521	71	0.8875	0.8796
2	.9970	.9953	37	.9565	.9507	72	.8850	.8771
3	.9956	.9938	38	.9550	.9489	73	.8825	.8746
4	.9942	.9922	39	.9535	.9473	74	.8799	.8719
5	.9930	.9909	40	.9519	.9456	75	.8769	.8689
6	.9914	.9893	41	.9503	.9438	76	.8745	.8665
7	.9898	.9876	42	.9490	.9424	77	.8721	.8641
8	.9890	.9868	43	.9470	.9402	78	.8696	.8616
9	.9878	.9855	44	.9452	.9382	79	.8664	.8583
10	.9869	.9846	45	.9434	.9363	80	.8639	.8558
11	.9855	.9831	46	.9416	.9343	81	.8611	.8530
12	.9841	.9816	47	.9396	.9323	82	.8581	.8500
13	.9828	.9801	48	.9381	.9307	83	.8557	.8476
14	.9821	.9793	49	.9362	.9288	84	.8526	.8444
15	.9815	.9787	50	.9343	.9267	85	.8496	.8414
16	.9802	.9773	51	.9323	.9246	86	.8466	.8384
17	.9789	.9759	52	.9303	.9226	87	.8434	.8352
18	.9778	.9746	53	.9283	.9205	88	.8408	.8326
19	.9766	.9733	54	.9262	.9184	89	.8373	.8291
20	.9760	.9726	55	.9242	.9164	90	.8340	.8258
21	.9753	.9719	56	.9221	.9143	91	.8305	.8223
22	.9741	.9706	57	.9200	.9122	92	.8272	.8191
23	.9728	.9692	58	.9178	.9100	93	.8237	.8156
24	.9716	.9678	59	.9160	.9081	94	.8199	.8118
25	.9709	.9668	60	.9135	.9056	95	.8164	.8083
26	.9698	.9655	61	.9113	.9034	96	.8125	.8044
27	.9691	.9646	62	.9090	.9011	97	.8084	.8003
28	.9678	.9631	63	.9069	.8989	98	.8041	.7960
29	.9665	.9617	64	.9047	.8969	99	.7995	.7914
30	.9652	.9603	65	.9025	.8947	100	.7946	.7865
31	.9643	.9594	66	.9001	.8923			
32	.9631	.9582	67	.8973	.8895			
33	.9618	.9567	68	.8949	.8870			
34	.9609	.9556	69	.8925	.8846			
35	.9593	.9538	70	.8900	.8821			

TABLE II.—*Percentage of alcohol.*

[Recalculated from the determinations of Gilpin, Drinkwater, and Squibb.]

Specific gravity at 60° F.	Alcohol.			Specific gravity at 60° F.	Alcohol.			Specific gravity at 60° F.	Alcohol.		
	Per cent by volume.	Per cent by weight.	Grams per 100 cc.		Per cent by volume.	Per cent by weight.	Grams per 100 cc.		Per cent by volume.	Per cent by weight.	Grams per 100 cc.
1.00000	0.00	0.00	0.00	0.99884	0.75	0.60	0.60	0.99775	1.50	1.19	1.19
0.99992	0.05	0.04	0.04	.99877	0.80	0.64	0.64	.99768	1.55	1.23	1.23
.99984	0.10	0.08	0.08	.99869	0.85	0.67	0.67	.99760	1.60	1.27	1.27
.99976	0.15	0.12	0.12	.99861	0.90	0.71	0.71	.99753	1.65	1.31	1.31
.99968	0.20	0.16	0.16	.99854	0.95	0.75	0.75	.99745	1.70	1.35	1.35
.99961	0.25	0.20	0.20	.99849	1.00	0.79	0.79	.99738	1.75	1.39	1.39
.99953	0.30	0.24	0.24	.99842	1.05	0.83	0.83	.99731	1.80	1.43	1.43
.99945	0.35	0.28	0.28	.99834	1.10	0.87	0.87	.99723	1.85	1.47	1.47
.99937	0.40	0.32	0.32	.99827	1.15	0.91	0.91	.99716	1.90	1.51	1.51
.99930	0.45	0.36	0.36	.99819	1.20	0.95	0.95	.99708	1.95	1.55	1.55
.99923	0.50	0.40	0.40	.99812	1.25	0.99	0.99	.99701	2.00	1.59	1.59
.99915	0.55	0.44	0.44	.99805	1.30	1.03	1.03	.99694	2.05	1.63	1.62
.99907	0.60	0.48	0.48	.99797	1.35	1.07	1.07	.99687	2.10	1.67	1.66
.99900	0.65	0.52	0.52	.99790	1.40	1.11	1.11	.99679	2.15	1.71	1.70
.99892	0.70	0.56	0.56	.99782	1.45	1.15	1.15	.99672	2.20	1.75	1.74

TABLE II.—Percentage of alcohol—Continued.

Specific gravity at 59° F.	Alcohol.			Specific gravity at 59° F.	Alcohol.			Specific gravity at 59° F.	Alcohol.		
	Per cent by volume.	Per cent by weight.	Grams per 100 cc.		Per cent by volume.	Per cent by weight.	Grams per 100 cc.		Per cent by volume.	Per cent by weight.	Grams per 100 cc.
0.99665	2.25	1.79	1.78	0.99215	5.50	4.40	4.37	0.98807	8.75	7.03	6.95
.99658	2.30	1.83	1.82	.99208	5.55	4.44	4.40	.98801	8.80	7.07	6.99
.99651	2.35	1.87	1.86	.99202	5.60	4.48	4.44	.98795	8.85	7.11	7.03
.99643	2.40	1.91	1.90	.99195	5.65	4.52	4.48	.98789	8.90	7.15	7.07
.99636	2.45	1.95	1.94	.99189	5.70	4.56	4.52	.98783	8.95	7.19	7.11
.99629	2.50	1.99	1.98	.99182	5.75	4.60	4.56	.98777	9.00	7.23	7.14
.99622	2.55	2.03	2.02	.99175	5.80	4.64	4.60	.98771	9.05	7.27	7.18
.99615	2.60	2.07	2.06	.99169	5.85	4.68	4.64	.98765	9.10	7.31	7.22
.99607	2.65	2.11	2.10	.99162	5.90	4.72	4.68	.98759	9.15	7.35	7.26
.99600	2.70	2.15	2.14	.99156	5.95	4.76	4.72	.98754	9.20	7.39	7.30
.99593	2.75	2.19	2.18	.99149	6.00	4.80	4.76	.98748	9.25	7.43	7.34
.99586	2.80	2.23	2.22	.99143	6.05	4.84	4.80	.98742	9.30	7.48	7.38
.99579	2.85	2.27	2.26	.99136	6.10	4.88	4.84	.98736	9.35	7.52	7.42
.99571	2.90	2.31	2.30	.99130	6.15	4.92	4.88	.98730	9.40	7.56	7.46
.99564	2.95	2.35	2.34	.99123	6.20	4.96	4.92	.98724	9.45	7.60	7.50
.99557	3.00	2.39	2.38	.99117	6.25	5.00	4.96	.98719	9.50	7.64	7.54
.99550	3.05	2.43	2.42	.99111	6.30	5.05	5.00	.98713	9.55	7.68	7.58
.99543	3.10	2.47	2.46	.99104	6.35	5.09	5.04	.98707	9.60	7.72	7.62
.99536	3.15	2.51	2.50	.99098	6.40	5.13	5.08	.98701	9.65	7.76	7.66
.99529	3.20	2.55	2.54	.99091	6.45	5.17	5.12	.98695	9.70	7.80	7.70
.99522	3.25	2.59	2.58	.99085	6.50	5.21	5.16	.98689	9.75	7.84	7.74
.99515	3.30	2.64	2.62	.99079	6.55	5.25	5.20	.98683	9.80	7.88	7.78
.99508	3.35	2.68	2.66	.99072	6.60	5.29	5.24	.98678	9.85	7.92	7.82
.99501	3.40	2.72	2.70	.99066	6.65	5.33	5.28	.98672	9.90	7.96	7.85
.99494	3.45	2.76	2.74	.99059	6.70	5.37	5.32	.98666	9.95	8.00	7.89
.99487	3.50	2.80	2.78	.99053	6.75	5.41	5.36	.98660	10.00	8.04	7.93
.99480	3.55	2.84	2.82	.99047	6.80	5.45	5.40	.98654	10.05	8.08	7.97
.99473	3.60	2.88	2.86	.99040	6.85	5.49	5.44	.98649	10.10	8.12	8.01
.99466	3.65	2.92	2.90	.99034	6.90	5.53	5.48	.98643	10.15	8.16	8.05
.99459	3.70	2.96	2.94	.99027	6.95	5.57	5.52	.98637	10.20	8.20	8.09
.99452	3.75	3.00	2.98	.99021	7.00	5.61	5.56	.98632	10.25	8.24	8.13
.99445	3.80	3.04	3.02	.99015	7.05	5.65	5.60	.98626	10.30	8.29	8.17
.99438	3.85	3.08	3.06	.99009	7.10	5.69	5.64	.98620	10.35	8.33	8.21
.99431	3.90	3.12	3.10	.99002	7.15	5.73	5.68	.98614	10.40	8.37	8.25
.99424	3.95	3.16	3.14	.98996	7.20	5.77	5.72	.98609	10.45	8.41	8.29
.99417	4.00	3.20	3.18	.98990	7.25	5.81	5.76	.98603	10.50	8.45	8.33
.99410	4.05	3.24	3.22	.98984	7.30	5.86	5.80	.98597	10.55	8.49	8.37
.99403	4.10	3.28	3.26	.98978	7.35	5.90	5.84	.98592	10.60	8.53	8.41
.99397	4.15	3.32	3.30	.98971	7.40	5.94	5.88	.98586	10.65	8.57	8.45
.99390	4.20	3.36	3.34	.98965	7.45	5.98	5.92	.98580	10.70	8.61	8.49
.99383	4.25	3.40	3.38	.98959	7.50	6.02	5.96	.98575	10.75	8.65	8.53
.99376	4.30	3.44	3.42	.98953	7.55	6.06	6.00	.98569	10.80	8.70	8.57
.99369	4.35	3.48	3.46	.98947	7.60	6.10	6.04	.98563	10.85	8.74	8.61
.99363	4.40	3.52	3.50	.98940	7.65	6.14	6.07	.98557	10.90	8.78	8.65
.99356	4.45	3.56	3.54	.98934	7.70	6.18	6.11	.98552	10.95	8.82	8.69
.99349	4.50	3.60	3.58	.98928	7.75	6.22	6.15	.98546	11.00	8.86	8.73
.99342	4.55	3.64	3.62	.98922	7.80	6.26	6.19	.98540	11.05	8.90	8.77
.99335	4.60	3.68	3.66	.98916	7.85	6.30	6.23	.98535	11.10	8.94	8.81
.99329	4.65	3.72	3.70	.98909	7.90	6.34	6.27	.98529	11.15	8.98	8.85
.99322	4.70	3.76	3.74	.98903	7.95	6.38	6.31	.98524	11.20	9.02	8.89
.99315	4.75	3.80	3.77	.98897	8.00	6.42	6.35	.98518	11.25	9.07	8.93
.99308	4.80	3.84	3.81	.98891	8.05	6.46	6.39	.98513	11.30	9.11	8.97
.99301	4.85	3.88	3.85	.98885	8.10	6.50	6.43	.98507	11.35	9.15	9.01
.99295	4.90	3.92	3.89	.98879	8.15	6.54	6.47	.98502	11.40	9.19	9.05
.99288	4.95	3.96	3.93	.98873	8.20	6.58	6.51	.98496	11.45	9.23	9.09
.99281	5.00	4.00	3.97	.98867	8.25	6.62	6.55	.98491	11.50	9.27	9.13
.99274	5.05	4.04	4.01	.98861	8.30	6.67	6.59	.98485	11.55	9.31	9.17
.99268	5.10	4.08	4.05	.98855	8.35	6.71	6.63	.98479	11.60	9.35	9.21
.99261	5.15	4.12	4.09	.98849	8.40	6.75	6.67	.98474	11.65	9.39	9.25
.99255	5.20	4.16	4.13	.98843	8.45	6.79	6.71	.98468	11.70	9.43	9.29
.99248	5.25	4.20	4.17	.98837	8.50	6.83	6.75	.98463	11.75	9.47	9.32
.99241	5.30	4.24	4.21	.98831	8.55	6.87	6.79	.98457	11.80	9.51	9.36
.99235	5.35	4.28	4.25	.98825	8.60	6.91	6.83	.98452	11.85	9.55	9.40
.99228	5.40	4.32	4.29	.98819	8.65	6.95	6.87	.98446	11.90	9.59	9.44
.99222	5.45	4.36	4.33	.98813	8.70	6.99	6.91	.98441	11.95	9.63	9.48

TABLE II.—Percentage of alcohol—Continued.

Specific gravity at 60° F.	Alcohol.			Specific gravity at 60° F.	Alcohol.			Specific gravity at 60° F.	Alcohol.		
	Per cent by volume.	Per cent by weight.	Grams per 100 cc.		Per cent by volume.	Per cent by weight.	Grams per 100 cc.		Per cent by volume.	Per cent by weight.	Grams per 100 cc.
0.98435	12.00	9.67	9.52	0.98088	15.25	12.33	12.10	0.97758	18.50	15.02	14.68
.98430	12.05	9.71	9.56	.98083	15.30	12.38	12.14	.97753	18.55	15.06	14.72
.98424	12.10	9.75	9.60	.98078	15.35	12.42	12.18	.97748	18.60	15.10	14.76
.98419	12.15	9.79	9.64	.98073	15.40	12.46	12.22	.97743	18.65	15.14	14.80
.98413	12.20	9.83	9.68	.98068	15.45	12.50	12.26	.97738	18.70	15.18	14.84
.98408	12.25	9.87	9.72	.98063	15.50	12.54	12.30	.97733	18.75	15.22	14.88
.98402	12.30	9.92	9.76	.98057	15.55	12.58	12.34	.97728	18.80	15.27	14.92
.98397	12.35	9.96	9.80	.98052	15.60	12.62	12.37	.97723	18.85	15.31	14.96
.98391	12.40	10.00	9.84	.98047	15.65	12.66	12.41	.97718	18.90	15.38	15.00
.98386	12.45	10.04	9.88	.98042	15.70	12.70	12.45	.97713	18.95	15.39	15.04
.98381	12.50	10.08	9.92	.98037	15.75	12.75	12.49	.97708	19.00	15.43	15.08
.98375	12.55	10.12	9.96	.98032	15.80	12.79	12.53	.97703	19.05	15.47	15.11
.98370	12.60	10.16	10.00	.98026	15.85	12.83	12.57	.97698	19.10	15.51	15.15
.98364	12.65	10.20	10.03	.98021	15.90	12.87	12.61	.97693	19.15	15.55	15.19
.98359	12.70	10.24	10.07	.98016	15.95	12.91	12.65	.97688	19.20	15.59	15.23
.98353	12.75	10.28	10.11	.98011	16.00	12.95	12.69	.97683	19.25	15.63	15.27
.98348	12.80	10.33	10.15	.98005	16.05	12.99	12.73	.97678	19.30	15.68	15.31
.98342	12.85	10.37	10.19	.98001	16.10	13.03	12.77	.97673	19.35	15.72	15.35
.98337	12.90	10.41	10.23	.97996	16.15	13.08	12.81	.97668	19.40	15.76	15.39
.98331	12.95	10.45	10.27	.97991	16.20	13.12	12.85	.97663	19.45	15.80	15.43
.98326	13.00	10.49	10.31	.97986	16.25	13.16	12.89	.97658	19.50	15.84	15.47
.98321	13.05	10.53	10.35	.97980	16.30	13.20	12.93	.97653	19.55	15.88	15.51
.98315	13.10	10.57	10.39	.97975	16.35	13.24	12.97	.97648	19.60	15.93	15.55
.98310	13.15	10.61	10.43	.97970	16.40	13.29	13.01	.97643	19.65	15.97	15.59
.98305	13.20	10.65	10.47	.97965	16.45	13.33	13.05	.97638	19.70	16.01	15.63
.98299	13.25	10.69	10.51	.97960	16.50	13.37	13.09	.97633	19.75	16.05	15.67
.98294	13.30	10.74	10.55	.97955	16.55	13.41	13.13	.97628	19.80	16.09	15.71
.98289	13.35	10.78	10.59	.97950	16.60	13.45	13.17	.97623	19.85	16.14	15.75
.98283	13.40	10.82	10.63	.97945	16.65	13.49	13.21	.97618	19.90	16.18	15.79
.98278	13.45	10.86	10.67	.97940	16.70	13.53	13.25	.97613	19.95	16.22	15.83
.98273	13.50	10.90	10.71	.97935	16.75	13.57	13.29	.97608	20.00	16.26	15.87
.98267	13.55	10.94	10.75	.97929	16.80	13.62	13.33	.97603	20.05	16.30	15.91
.98262	13.60	10.98	10.79	.97924	16.85	13.66	13.37	.97598	20.10	16.34	15.95
.98256	13.65	11.02	10.83	.97919	16.90	13.70	13.41	.97593	20.15	16.38	15.99
.98251	13.70	11.06	10.87	.97914	16.95	13.74	13.45	.97588	20.20	16.42	16.03
.98246	13.75	11.11	10.91	.97909	17.00	13.78	13.49	.97583	20.25	16.46	16.06
.98240	13.80	11.15	10.95	.97904	17.05	13.82	13.53	.97578	20.30	16.51	16.10
.98235	13.85	11.19	10.99	.97899	17.10	13.86	13.57	.97573	20.35	16.58	16.14
.98230	13.90	11.23	11.03	.97894	17.15	13.90	13.61	.97568	20.40	16.59	16.18
.98224	13.95	11.27	11.07	.97889	17.20	13.94	13.65	.97563	20.45	16.63	16.22
.98219	14.00	11.31	11.11	.97884	17.25	13.98	13.69	.97558	20.50	16.67	16.26
.98214	14.05	11.35	11.15	.97879	17.30	14.03	13.73	.97553	20.55	16.71	16.30
.98209	14.10	11.39	11.19	.97874	17.35	14.07	13.77	.97548	20.60	16.75	16.34
.98203	14.15	11.43	11.23	.97869	17.40	14.11	13.81	.97543	20.65	16.80	16.38
.98198	14.20	11.47	11.27	.97864	17.45	14.15	13.85	.97538	20.70	16.84	16.42
.98193	14.25	11.52	11.31	.97859	17.50	14.19	13.89	.97533	20.75	16.88	16.46
.98188	14.30	11.56	11.35	.97853	17.55	14.23	13.92	.97528	20.80	16.92	16.50
.98182	14.35	11.60	11.39	.97848	17.60	14.27	13.96	.97523	20.85	16.96	16.54
.98177	14.40	11.64	11.43	.97843	17.65	14.31	14.00	.97518	20.90	17.01	16.58
.98172	14.45	11.68	11.47	.97838	17.70	14.35	14.04	.97513	20.95	17.05	16.62
.98167	14.50	11.72	11.51	.97833	17.75	14.40	14.08	.97508	21.00	17.09	16.66
.98161	14.55	11.76	11.55	.97828	17.80	14.44	14.12	.97503	21.05	17.13	16.70
.98156	14.60	11.80	11.59	.97823	17.85	14.48	14.16	.97498	21.10	17.17	16.74
.98151	14.65	11.84	11.63	.97818	17.90	14.52	14.20	.97493	21.15	17.22	16.78
.98146	14.70	11.88	11.67	.97813	17.95	14.56	14.24	.97488	21.20	17.26	16.82
.98140	14.75	11.93	11.71	.97808	18.00	14.60	14.28	.97483	21.25	17.30	16.86
.98135	14.80	11.97	11.75	.97803	18.05	14.64	14.32	.97478	21.30	17.34	16.90
.98130	14.85	12.01	11.79	.97798	18.10	14.68	14.36	.97473	21.35	17.38	16.94
.98125	14.90	12.05	11.82	.97793	18.15	14.73	14.40	.97468	21.40	17.43	16.98
.98119	14.95	12.09	11.86	.97788	18.20	14.77	14.44	.97463	21.45	17.47	17.02
.98114	15.00	12.13	11.90	.97783	18.25	14.81	14.48	.97458	21.50	17.51	17.06
.98108	15.05	12.17	11.94	.97778	18.30	14.85	14.52	.97453	21.55	17.55	17.10
.98104	15.10	12.21	11.98	.97773	18.35	14.89	14.56	.97448	21.60	17.59	17.14
.98099	15.15	12.25	12.02	.97768	18.40	14.94	14.60	.97443	21.65	17.63	17.18
.98093	15.20	12.29	12.06	.97763	18.45	14.98	14.64	.97438	21.70	17.67	17.22

TABLE II.—Percentage of alcohol—Continued.

Specific gravity at 60° F.	Alcohol.			Specific gravity at 60° F.	Alcohol.			Specific gravity at 60° F.	Alcohol.		
	Per cent by volume.	Per cent by weight.	Grams per 100 cc.		Per cent by volume.	Per cent by weight.	Grams per 100 cc.		Per cent by volume.	Per cent by weight.	Grams per 100 cc.
0.97431	21.75	17.71	17.26	0.97097	25.00	20.43	19.84	0.96744	28.25	23.17	22.42
.97426	21.80	17.76	17.30	.97092	25.05	20.47	19.88	.96738	28.30	23.21	22.45
.97421	21.85	17.80	17.34	.97086	25.10	20.51	19.92	.96732	28.35	23.25	22.49
.97416	21.90	17.84	17.38	.97081	25.15	20.56	19.96	.96726	28.40	23.30	22.53
.97411	21.95	17.88	17.42	.97076	25.20	20.60	20.00	.96721	28.45	23.34	22.57
.97406	22.00	17.92	17.46	.97071	25.25	20.64	20.04	.96715	28.50	23.38	22.61
.97401	22.05	17.96	17.50	.97065	25.30	20.68	20.08	.96709	28.55	23.42	22.65
.97396	22.10	18.00	17.54	.97060	25.35	20.72	20.12	.96704	28.60	23.47	22.69
.97391	22.15	18.05	17.58	.97055	25.40	20.77	20.16	.96698	28.65	23.51	22.73
.97386	22.20	18.09	17.62	.97049	25.45	20.81	20.20	.96692	28.70	23.55	22.77
.97381	22.25	18.13	17.66	.97044	25.50	20.85	20.24	.96687	28.75	23.60	22.81
.97375	22.30	18.17	17.70	.97039	25.55	20.89	20.28	.96681	28.80	23.64	22.85
.97370	22.35	18.21	17.74	.97033	25.60	20.93	20.32	.96675	28.85	23.68	22.89
.97365	22.40	18.26	17.78	.97028	25.65	20.98	20.36	.96669	28.90	23.72	22.93
.97360	22.45	18.30	17.82	.97023	25.70	21.02	20.40	.96664	28.95	23.77	22.97
.97355	22.50	18.34	17.86	.97018	25.75	21.06	20.44	.96658	29.00	23.81	23.01
.97350	22.55	18.38	17.90	.97012	25.80	21.10	20.47	.96652	29.05	23.85	23.05
.97345	22.60	18.42	17.94	.97007	25.85	21.14	20.51	.96646	29.10	23.89	23.09
.97340	22.65	18.47	17.98	.97001	25.90	21.19	20.55	.96640	29.15	23.94	23.13
.97335	22.70	18.51	18.02	.96996	25.95	21.23	20.59	.96635	29.20	23.98	23.17
.97330	22.75	18.55	18.06	.96991	26.00	21.27	20.63	.96629	29.25	24.02	23.21
.97324	22.80	18.59	18.10	.96986	26.05	21.31	20.67	.96623	29.30	24.06	23.25
.97319	22.85	18.63	18.14	.96980	26.10	21.35	20.71	.96617	29.35	24.10	23.29
.97314	22.90	18.68	18.18	.96975	26.15	21.40	20.75	.96611	29.40	24.15	23.33
.97309	22.95	18.72	18.22	.96969	26.20	21.44	20.79	.96605	29.45	24.19	23.37
.97304	23.00	18.76	18.26	.96964	26.25	21.48	20.83	.96600	29.50	24.23	23.41
.97299	23.05	18.80	18.29	.96959	26.30	21.52	20.87	.96594	29.55	24.27	23.45
.97294	23.10	18.84	18.33	.96953	26.35	21.56	20.91	.96588	29.60	24.32	23.49
.97289	23.15	18.88	18.37	.96949	26.40	21.61	20.95	.96582	29.65	24.36	23.53
.97283	23.20	18.92	18.41	.96942	26.45	21.65	20.99	.96576	29.70	24.40	23.57
.97278	23.25	18.96	18.45	.96937	26.50	21.69	21.03	.96570	29.75	24.45	23.61
.97273	23.30	19.01	18.49	.96932	26.55	21.73	21.07	.96564	29.80	24.49	23.65
.97268	23.35	19.05	18.53	.96926	26.60	21.77	21.11	.96559	29.85	24.53	23.69
.97263	23.40	19.09	18.57	.96921	26.65	21.82	21.15	.96553	29.90	24.57	23.73
.97258	23.45	19.13	18.61	.96915	26.70	21.86	21.19	.96547	29.95	24.62	23.77
.97253	23.50	19.17	18.65	.96910	26.75	21.90	21.23	.96541	30.00	24.66	23.81
.97247	23.55	19.21	18.69	.96905	26.80	21.94	21.27	.96535	30.05	24.70	23.85
.97242	23.60	19.25	18.73	.96899	26.85	21.98	21.31	.96529	30.10	24.74	23.89
.97237	23.65	19.30	18.77	.96894	26.90	22.03	21.35	.96523	30.15	24.79	23.93
.97232	23.70	19.34	18.81	.96888	26.95	22.07	21.39	.96517	30.20	24.83	23.97
.97227	23.75	19.38	18.84	.96883	27.00	22.11	21.43	.96511	30.25	24.87	24.01
.97222	23.80	19.42	18.88	.96877	27.05	22.15	21.47	.96505	30.30	24.91	24.04
.97216	23.85	19.46	18.92	.96872	27.10	22.20	21.51	.96499	30.35	24.95	24.08
.97211	23.90	19.51	18.96	.96866	27.15	22.24	21.55	.96493	30.40	25.00	24.12
.97206	23.95	19.55	19.00	.96861	27.20	22.28	21.59	.96487	30.45	25.04	24.16
.97201	24.00	19.59	19.04	.96855	27.25	22.33	21.63	.96481	30.50	25.08	24.20
.97196	24.05	19.63	19.08	.96850	27.30	22.37	21.67	.96475	30.55	25.12	24.24
.97191	24.10	19.67	19.12	.96844	27.35	22.41	21.71	.96469	30.60	25.17	24.28
.97185	24.15	19.72	19.16	.96839	27.40	22.45	21.75	.96463	30.65	25.21	24.32
.97180	24.20	19.76	19.20	.96833	27.45	22.50	21.79	.96457	30.70	25.25	24.36
.97175	24.25	19.80	19.24	.96828	27.50	22.54	21.83	.96451	30.75	25.30	24.40
.97170	24.30	19.84	19.28	.96822	27.55	22.58	21.86	.96445	30.80	25.34	24.44
.97165	24.35	19.88	19.32	.96816	27.60	22.62	21.90	.96439	30.85	25.38	24.48
.97159	24.40	19.93	19.36	.96811	27.65	22.67	21.94	.96433	30.90	25.42	24.52
.97154	24.45	19.97	19.40	.96805	27.70	22.71	21.98	.96427	30.95	25.47	24.56
.97149	24.50	20.01	19.44	.96800	27.75	22.75	22.02	.96421	31.00	25.51	24.60
.97144	24.55	20.05	19.48	.96794	27.80	22.79	22.06	.96415	31.05	25.55	24.64
.97139	24.60	20.09	19.52	.96789	27.85	22.83	22.10	.96409	31.10	25.60	24.68
.97133	24.65	20.14	19.56	.96783	27.90	22.88	22.14	.96403	31.15	25.64	24.72
.97128	24.70	20.18	19.60	.96778	27.95	22.92	22.18	.96396	31.20	25.68	24.76
.97123	24.75	20.22	19.64	.96772	28.00	22.96	22.22	.96390	31.25	25.73	24.80
.97118	24.80	20.26	19.68	.96766	28.05	23.00	22.26	.96384	31.30	25.77	24.84
.97113	24.85	20.30	19.72	.96761	28.10	23.04	22.30	.96378	31.35	25.81	24.88
.97107	24.90	20.35	19.76	.96755	28.15	23.09	22.34	.96372	31.40	25.85	24.92
.97102	24.95	20.39	19.80	.96749	28.20	23.13	22.38	.96366	31.45	25.90	24.96

TABLE II.—Percentage of alcohol—Continued.

Specific gravity at 60° F.	Alcohol.			Specific gravity at 60° F.	Alcohol.			Specific gravity at 60° F.	Alcohol.		
	Per cent by volume.	Per cent by weight.	Grams per 100 cc.		Per cent by volume.	Per cent by weight.	Grams per 100 cc.		Per cent by volume.	Per cent by weight.	Grams per 100 cc.
0.96360	31.50	25.94	25.00	0.95943	34.75	28.74	27.58	0.95487	38.00	31.58	30.16
.96353	31.55	25.98	25.04	.95937	34.80	28.78	27.62	.95480	38.05	31.63	30.20
.96347	31.60	26.03	25.08	.95930	34.85	28.83	27.66	.95472	38.10	31.67	30.24
.96341	31.65	26.07	25.12	.95923	34.90	28.87	27.70	.95465	38.15	31.72	30.28
.96335	31.70	26.11	25.16	.95917	34.95	28.92	27.74	.95457	38.20	31.76	30.32
.96329	31.75	26.16	25.20	.95910	35.00	28.96	27.78	.95450	38.25	31.81	30.36
.96323	31.80	26.20	25.24	.95903	35.05	29.00	27.82	.95442	38.30	31.85	30.40
.96316	31.85	26.24	25.28	.95896	35.10	29.05	27.86	.95435	38.35	31.90	30.44
.96310	31.90	26.28	25.32	.95889	35.15	29.09	27.90	.95427	38.40	31.94	30.48
.96304	31.95	26.33	25.36	.95883	35.20	29.13	27.94	.95420	38.45	31.99	30.52
.96298	32.00	26.37	25.40	.95876	35.25	29.18	27.98	.95413	38.50	32.03	30.56
.96292	32.05	26.41	25.44	.95869	35.30	29.22	28.02	.95405	38.55	32.07	30.60
.96285	32.10	26.46	25.48	.95862	35.35	29.26	28.05	.95398	38.60	32.12	30.64
.96279	32.15	26.50	25.52	.95855	35.40	29.30	28.09	.95390	38.65	32.16	30.68
.96273	32.20	26.54	25.56	.95848	35.45	29.35	28.13	.95383	38.70	32.20	30.72
.96267	32.25	26.59	25.60	.95842	35.50	29.30	28.17	.95375	38.75	32.25	30.76
.96260	32.30	26.63	25.64	.95835	35.55	29.43	28.21	.95368	38.80	32.29	30.79
.96254	32.35	26.67	25.68	.95828	35.60	29.48	28.25	.95360	38.85	32.33	30.83
.96248	32.40	26.71	25.71	.95821	35.65	29.52	28.29	.95353	38.90	32.37	30.87
.96241	32.45	26.76	25.75	.95814	35.70	29.57	28.33	.95345	38.95	32.42	30.91
.96235	32.50	26.80	25.79	.95807	35.75	29.61	28.37	.95338	39.00	32.46	30.95
.96229	32.55	26.84	25.83	.95800	35.80	29.65	28.41	.95330	39.05	32.50	30.99
.96222	32.60	26.89	25.87	.95794	35.85	29.70	28.45	.95323	39.10	32.55	31.03
.96216	32.65	26.93	25.91	.95787	35.90	29.74	28.49	.95315	39.15	32.59	31.07
.96210	32.70	26.97	25.95	.95780	35.95	29.79	28.53	.95307	39.20	32.64	31.11
.96204	32.75	27.02	25.99	.95773	36.00	29.83	28.57	.95300	39.25	32.68	31.14
.96197	32.80	27.06	26.03	.95766	36.05	29.87	28.61	.95292	39.30	32.72	31.18
.96191	32.85	27.10	26.07	.95759	36.10	29.92	28.65	.95284	39.35	32.77	31.22
.96185	32.90	27.14	26.11	.95752	36.15	29.96	28.69	.95277	39.40	32.81	31.26
.96178	32.95	27.19	26.15	.95745	36.20	30.00	28.73	.95269	39.45	32.86	31.30
.96172	33.00	27.23	26.19	.95738	36.25	30.05	28.77	.95262	39.50	32.90	31.34
.96166	33.05	27.27	26.23	.95731	36.30	30.09	28.81	.95254	39.55	32.95	31.38
.96159	33.10	27.32	26.27	.95724	36.35	30.13	28.84	.95246	39.60	32.99	31.42
.96153	33.15	27.36	26.31	.95717	36.40	30.17	28.88	.95239	39.65	33.04	31.46
.96146	33.20	27.40	26.35	.95710	36.45	30.22	28.92	.95231	39.70	33.08	31.50
.96140	33.25	27.45	26.39	.95703	36.50	30.26	28.96	.95223	39.75	33.13	31.54
.96133	33.30	27.49	26.43	.95696	36.55	30.30	29.00	.95216	39.80	33.17	31.58
.96127	33.35	27.53	26.47	.95688	36.60	30.35	29.04	.95208	39.85	33.22	31.62
.96120	33.40	27.57	26.51	.95681	36.65	30.39	29.08	.95200	39.90	33.27	31.66
.96114	33.45	27.62	26.55	.95674	36.70	30.44	29.12	.95193	39.95	33.31	31.70
.96108	33.50	27.66	26.59	.95667	36.75	30.48	29.16	.95185	40.00	33.35	31.74
.96101	33.55	27.70	26.63	.95660	36.80	30.52	29.20	.95177	40.05	33.39	31.78
.96095	33.60	27.75	26.67	.95653	36.85	30.57	29.24	.95169	40.10	33.44	31.82
.96088	33.65	27.79	26.71	.95646	36.90	30.61	29.29	.95161	40.15	33.48	31.86
.96082	33.70	27.83	26.75	.95639	36.95	30.66	29.32	.95154	40.20	33.53	31.90
.96075	33.75	27.88	26.79	.95632	37.00	30.70	29.36	.95146	40.25	33.57	31.94
.96069	33.80	27.92	26.82	.95625	37.05	30.74	29.40	.95138	40.30	33.61	31.98
.96062	33.85	27.96	26.86	.95618	37.10	30.79	29.44	.95130	40.35	33.66	32.02
.96056	33.90	28.00	26.90	.95610	37.15	30.83	29.48	.95122	40.40	33.70	32.06
.96049	33.95	28.05	26.94	.95603	37.20	30.88	29.52	.95114	40.45	33.75	32.10
.96043	34.00	28.09	26.98	.95596	37.25	30.92	29.56	.95107	40.50	33.79	32.14
.96036	34.05	28.13	27.02	.95589	37.30	30.96	29.60	.95099	40.55	33.84	32.18
.96030	34.10	28.18	27.06	.95581	37.35	31.01	29.64	.95091	40.60	33.88	32.22
.96023	34.15	28.22	27.10	.95574	37.40	31.05	29.68	.95083	40.65	33.93	32.26
.96016	34.20	28.26	27.14	.95567	37.45	31.10	29.72	.95075	40.70	33.97	32.30
.96010	34.25	28.31	27.18	.95560	37.50	31.14	29.76	.95067	40.75	34.02	32.34
.96003	34.30	28.35	27.22	.95552	37.55	31.18	29.80	.95059	40.80	34.06	32.38
.95996	34.35	28.39	27.26	.95545	37.60	31.23	29.84	.95052	40.85	34.11	32.42
.95990	34.40	28.43	27.30	.95538	37.65	31.27	29.88	.95044	40.90	34.15	32.46
.95983	34.45	28.48	27.34	.95531	37.70	31.32	29.92	.95036	40.95	34.20	32.50
.95977	34.50	28.52	27.38	.95523	37.75	31.36	29.96	.95028	41.00	34.24	32.54
.95970	34.55	28.56	27.42	.95516	37.80	31.40	30.00	.95020	41.05	34.28	32.58
.95963	34.60	28.61	27.46	.95509	37.85	31.45	30.04	.95012	41.10	34.33	32.62
.95957	34.65	28.65	27.50	.95502	37.90	31.49	30.08	.95004	41.15	34.37	32.66
.95950	34.70	28.70	27.54	.95494	37.95	31.54	30.12	.94996	41.20	34.42	32.70

TABLE II.—Percentage of alcohol—Continued.

Specific gravity at 60° F.	Alcohol.			Specific gravity at 60° F.	Alcohol.			Specific gravity at 60° F.	Alcohol.		
	Per cent by vol-ume.	Per cent by weight.	Grams per 100 cc.		Per cent by vol-ume.	Per cent by weight.	Grams per 100 cc.		Per cent by vol-ume.	Per cent by weight.	Grams per 100 cc.
0.94988	41.25	34.46	32.74	0.94493	44.25	37.16	35.11	0.93962	47.25	39.90	37.49
.94980	41.30	34.50	32.78	.94484	44.30	37.21	35.15	.93953	47.30	39.95	37.53
.94972	41.35	34.55	32.82	.94476	44.35	37.25	35.19	.93944	47.35	39.99	37.57
.94964	41.40	34.59	32.86	.94467	44.40	37.30	35.23	.93934	47.40	40.04	37.61
.94956	41.45	34.64	32.90	.94459	44.45	37.34	35.27	.93925	47.45	40.08	37.65
.94948	41.50	34.68	32.93	.94450	44.50	37.39	35.31	.93916	47.50	40.13	37.69
.94940	41.55	34.73	32.97	.94441	44.55	37.44	35.35	.93906	47.55	40.18	37.73
.94932	41.60	34.77	33.01	.94433	44.60	37.48	35.39	.93898	47.60	40.22	37.77
.94924	41.65	34.82	33.05	.94424	44.65	37.53	35.43	.93888	47.65	40.27	37.81
.94916	41.70	34.86	33.09	.94416	44.70	37.57	35.47	.93879	47.70	40.32	37.85
.94908	41.75	34.91	33.13	.94407	44.75	37.62	35.51	.93870	47.75	40.37	37.89
.94900	41.80	34.95	33.17	.94398	44.80	37.66	35.55	.93861	47.80	40.41	37.93
.94892	41.85	35.00	33.21	.94390	44.85	37.71	35.59	.93852	47.85	40.46	37.97
.94884	41.90	35.04	33.25	.94381	44.90	37.76	35.63	.93842	47.90	40.51	38.01
.94876	41.95	35.09	33.29	.94373	44.95	37.80	35.67	.93833	47.95	40.55	38.05
.94868	42.00	35.13	33.33	.94364	45.00	37.84	35.71	.93824	48.00	40.60	38.09
.94860	42.05	35.18	33.37	.94356	45.05	37.89	35.75	.93815	48.05	40.65	38.13
.94852	42.10	35.22	33.41	.94346	45.10	37.93	35.79	.93805	48.10	40.69	38.17
.94843	42.15	35.27	33.45	.94338	45.15	37.98	35.83	.93796	48.15	40.74	38.21
.94835	42.20	35.31	33.49	.94329	45.20	38.02	35.87	.93786	48.20	40.78	38.25
.94827	42.25	35.36	33.53	.94320	45.25	38.07	35.91	.93777	48.25	40.83	38.29
.94810	42.30	35.40	33.57	.94311	45.30	38.12	35.95	.93768	48.30	40.88	38.33
.94811	42.35	35.45	33.61	.94302	45.35	38.16	35.99	.93758	48.35	40.92	38.37
.94802	42.40	35.49	33.65	.94294	45.40	38.21	36.03	.93749	48.40	40.97	38.41
.94794	42.45	35.54	33.69	.94285	45.45	38.25	36.07	.93739	48.45	41.01	38.45
.94786	42.50	35.58	33.73	.94276	45.50	38.30	36.11	.93730	48.50	41.06	38.49
.94778	42.55	35.63	33.77	.94267	45.55	38.35	36.15	.93721	48.55	41.11	38.53
.94770	42.60	35.67	33.81	.94258	45.60	38.39	36.19	.93711	48.60	41.15	38.57
.94761	42.65	35.72	33.85	.94250	45.65	38.44	36.23	.93702	48.65	41.20	38.61
.94753	42.70	35.76	33.89	.94241	45.70	38.48	36.26	.93692	48.70	41.24	38.65
.94745	42.75	35.81	33.93	.94232	45.75	38.53	36.30	.93683	48.75	41.29	38.68
.94737	42.80	35.85	33.97	.94223	45.80	38.57	36.34	.93674	48.80	41.34	38.72
.94729	42.85	35.90	34.00	.94214	45.85	38.62	36.38	.93664	48.85	41.38	38.76
.94720	42.90	35.94	34.04	.94206	45.90	38.66	36.42	.93655	48.90	41.43	38.80
.94712	42.95	35.99	34.08	.94197	45.95	38.71	36.46	.93645	48.95	41.47	38.84
.94704	43.00	36.03	34.12	.94188	46.00	38.75	36.50	.93636	49.00	41.52	38.88
.94696	43.05	36.08	34.16	.94179	46.05	38.80	36.54	.93626	49.05	41.57	38.92
.94687	43.10	36.12	34.20	.94170	46.10	38.84	36.58	.93617	49.10	41.61	38.96
.94679	43.15	36.17	34.24	.94161	46.15	38.89	36.62	.93607	49.15	41.66	39.00
.94670	43.20	36.21	34.28	.94152	46.20	38.93	36.66	.93598	49.20	41.71	39.04
.94662	43.25	36.23	34.32	.94143	46.25	38.98	36.70	.93588	49.25	41.76	39.08
.94654	43.30	36.30	34.36	.94134	46.30	39.03	36.74	.93578	49.30	41.80	39.12
.94645	43.35	36.35	34.40	.94125	46.35	39.07	36.78	.93569	49.35	41.85	39.16
.94637	43.40	36.39	34.44	.94116	46.40	39.12	36.82	.93559	49.40	41.90	39.20
.94628	43.45	36.44	34.48	.94107	46.45	39.16	36.86	.93550	49.45	41.94	39.24
.94620	43.50	36.48	34.52	.94098	46.50	39.21	36.90	.93540	49.50	41.99	39.28
.94612	43.55	36.53	34.56	.94089	46.55	39.26	36.94	.93530	49.55	42.04	39.32
.94603	43.60	36.57	34.60	.94080	46.60	39.30	36.98	.93521	49.60	42.08	39.36
.94595	43.65	36.62	34.64	.94071	46.65	39.35	37.02	.93511	49.65	42.13	39.40
.94586	43.70	36.66	34.68	.94062	46.70	39.39	37.06	.93502	49.70	42.18	39.44
.94578	43.75	36.71	34.72	.94053	46.75	39.44	37.09	.93492	49.75	42.23	39.48
.94570	43.80	36.75	34.76	.94044	46.80	39.49	37.13	.93482	49.80	42.27	39.52
.94561	43.85	36.80	34.80	.94035	46.85	39.53	37.17	.93473	49.85	42.32	39.56
.94553	43.90	36.84	34.84	.94026	46.90	39.58	37.21	.93463	49.90	42.37	39.60
.94544	43.95	36.89	34.88	.94017	46.95	39.62	37.25	.93454	49.95	42.41	39.63
.94536	44.00	36.93	34.91	.94008	47.00	39.67	37.29				
.94527	44.05	36.98	34.95	.93999	47.05	39.72	37.33				
.94519	44.10	37.02	34.99	.93990	47.10	39.76	37.37				
.94510	44.15	37.07	35.03	.93980	47.15	39.81	37.41				
.94502	44.20	37.11	35.07	.93971	47.20	39.85	37.45				



TABLE III.—*Extract in beer wort.\**

[According to Schultz and Ostermann.]

Specific gravity at 15°C.	Extract.		Specific gravity at 15°C.	Extract.		Specific gravity at 15°C.	Extract.		Specific gravity at 15°C.	Extract.	
	Per cent by weight.	Grams per 100 cc.		Per cent by weight.	Grams per 100 cc.		Per cent by weight.	Grams per 100 cc.		Per cent by weight.	Grams per 100 cc.
1.0000	0.00	0.00	1.0065	1.69	1.70	1.0130	3.35	3.39	1.0195	5.06	5.16
1.0001	0.03	0.03	1.0066	1.72	1.73	1.0131	3.38	3.42	1.0196	5.09	5.19
1.0002	0.05	0.05	1.0067	1.74	1.75	1.0132	3.41	3.46	1.0197	5.12	5.22
1.0003	0.08	0.08	1.0068	1.77	1.78	1.0133	3.43	3.48	1.0198	5.15	5.25
1.0004	0.10	1.10	1.0069	1.79	1.80	1.0134	3.46	3.51	1.0199	5.17	5.27
1.0005	0.13	0.13	1.0070	1.82	1.83	1.0135	3.48	3.53	1.0200	5.20	5.30
1.0006	0.16	0.16	1.0071	1.84	1.85	1.0136	3.51	3.56	1.0201	5.23	5.34
1.0007	0.18	0.18	1.0072	1.87	1.88	1.0127	3.54	3.59	1.0202	5.25	5.36
1.0008	0.21	0.21	1.0073	1.90	1.91	1.0138	3.56	3.61	1.0203	5.28	5.39
1.0009	0.24	0.24	1.0074	1.92	1.93	1.0139	3.59	3.64	1.0204	5.30	5.41
1.0010	0.26	0.26	1.0075	1.95	1.96	1.0140	3.61	3.66	1.0205	5.33	5.44
1.0011	0.29	0.29	1.0076	1.97	1.98	1.0141	3.64	3.69	1.0206	5.35	5.46
1.0012	0.31	0.31	1.0077	2.00	2.02	1.0142	3.66	3.71	1.0207	5.38	5.49
1.0013	0.34	0.34	1.0078	2.02	2.04	1.0143	3.69	3.74	1.0208	5.40	5.51
1.0014	0.37	0.37	1.0079	2.05	2.07	1.0144	3.72	4.77	1.0209	5.43	5.54
1.0015	0.39	0.39	1.0080	2.07	2.09	1.0145	3.74	3.79	1.0210	5.45	5.56
1.0016	0.42	0.42	1.0081	2.10	2.12	1.0146	3.77	3.83	1.0211	5.48	5.60
1.0017	0.45	0.45	1.0082	2.12	2.14	1.0147	3.79	3.85	1.0212	5.50	5.62
1.0018	0.47	0.47	1.0083	2.15	2.17	1.0148	3.82	3.88	1.0213	5.53	5.65
1.0019	0.50	0.50	1.0084	2.17	2.19	1.0149	3.85	3.91	1.0214	5.55	5.67
1.0020	0.52	0.52	1.0085	2.20	2.22	1.0150	3.87	3.93	1.0215	5.57	5.69
1.0021	0.55	0.55	1.0086	2.23	2.25	1.0151	3.90	3.96	1.0216	5.60	5.72
1.0022	0.58	0.58	1.0087	2.25	2.27	1.0152	3.92	3.98	1.0217	5.62	5.74
1.0023	0.60	0.60	1.0088	2.28	2.30	1.0153	3.95	4.01	1.0218	5.65	5.77
1.0024	0.63	0.63	1.0089	2.30	2.32	1.0154	3.97	4.03	1.0219	5.67	5.79
1.0025	0.66	0.66	1.0090	2.33	2.35	1.0155	4.00	4.06	1.0220	5.70	5.83
1.0026	0.68	0.68	1.0091	2.35	2.37	1.0156	4.03	4.09	1.0221	5.72	5.85
1.0027	0.71	0.71	1.0092	2.38	2.40	1.0157	4.05	4.11	1.0222	5.75	5.88
1.0028	0.73	0.73	1.0093	2.41	2.43	1.0158	4.08	4.14	1.0223	5.77	5.90
1.0029	0.76	0.76	1.0094	2.43	2.45	1.0159	4.10	4.17	1.0224	5.80	5.93
1.0030	0.79	0.79	1.0095	2.46	2.48	1.0160	4.13	4.20	1.0225	5.82	5.95
1.0031	0.81	0.81	1.0096	2.48	2.50	1.0161	4.16	4.23	1.0226	5.84	5.97
1.0032	0.84	0.84	1.0097	2.51	2.53	1.0162	4.18	4.25	1.0227	5.87	6.00
1.0033	0.87	0.87	1.0098	2.53	2.55	1.0163	4.21	4.28	1.0228	5.89	6.02
1.0034	0.89	0.89	1.0099	2.56	2.59	1.0164	4.23	4.30	1.0229	5.92	6.06
1.0035	0.92	0.92	1.0100	2.58	2.61	1.0165	4.26	4.33	1.0230	5.94	6.08
1.0036	0.94	0.94	1.0101	2.61	2.64	1.0166	4.28	4.35	1.0231	5.97	6.11
1.0037	0.97	0.97	1.0102	2.64	2.67	1.0167	4.31	4.38	1.0232	5.99	6.13
1.0038	1.00	1.00	1.0103	2.66	2.69	1.0168	4.34	4.41	1.0233	6.02	6.16
1.0039	1.02	1.02	1.0104	2.69	2.72	1.0169	4.36	4.43	1.0234	6.04	6.18
1.0040	1.05	1.05	1.0105	2.71	2.74	1.0170	4.39	4.46	1.0235	6.07	6.21
1.0041	1.08	1.08	1.0106	2.74	2.77	1.0171	4.42	4.50	1.0236	6.09	6.23
1.0042	1.10	1.10	1.0107	2.76	2.79	1.0172	4.44	4.52	1.0237	6.11	6.25
1.0043	1.13	1.13	1.0108	2.79	2.82	1.0173	4.47	4.55	1.0238	6.14	6.29
1.0044	1.15	1.16	1.0109	2.82	2.85	1.0174	4.50	4.58	1.0239	6.16	6.31
1.0045	1.18	1.19	1.0110	2.84	2.87	1.0175	4.53	4.61	1.0240	6.19	6.34
1.0046	1.21	1.22	1.0111	2.87	2.90	1.0176	4.55	4.63	1.0241	6.21	6.36
1.0047	1.23	1.24	1.0112	2.89	2.92	1.0177	4.58	4.66	1.0242	6.24	6.39
1.0048	1.26	1.27	1.0113	2.92	2.95	1.0178	4.61	4.69	1.0243	6.26	6.41
1.0049	1.29	1.30	1.0114	2.94	2.97	1.0179	4.63	4.71	1.0244	6.29	6.44
1.0050	1.31	1.32	1.0115	2.97	3.00	1.0180	4.66	4.74	1.0245	6.31	6.46
1.0051	1.34	1.35	1.0116	2.99	3.02	1.0181	4.69	4.77	1.0246	6.34	6.50
1.0052	1.36	1.37	1.0117	3.02	3.06	1.0182	4.71	4.80	1.0247	6.36	6.52
1.0053	1.39	1.40	1.0118	3.05	3.09	1.0183	4.74	4.83	1.0248	6.39	6.55
1.0054	1.41	1.42	1.0119	3.07	3.11	1.0184	4.77	4.86	1.0249	6.41	6.57
1.0055	1.44	1.45	1.0120	3.10	3.14	1.0185	4.79	4.88	1.0250	6.44	6.60
1.0056	1.46	1.47	1.0121	3.12	3.16	1.0186	4.82	4.91	1.0251	6.47	6.63
1.0057	1.49	1.50	1.0122	3.15	3.19	1.0187	4.85	4.94	1.0252	6.50	6.66
1.0058	1.51	1.52	1.0123	3.17	3.21	1.0188	4.88	4.97	1.0253	6.52	6.68
1.0059	1.54	1.55	1.0124	3.20	3.24	1.0189	4.90	4.99	1.0254	6.55	6.72
1.0060	1.56	1.57	1.0125	3.23	3.27	1.0190	4.93	5.02	1.0255	6.58	6.75
1.0061	1.59	1.60	1.0126	3.25	3.29	1.0191	4.96	5.05	1.0256	6.61	6.78
1.0062	1.62	1.63	1.0127	3.28	3.32	1.0192	4.98	5.08	1.0257	6.63	6.80
1.0063	1.64	1.65	1.0128	3.30	3.34	1.0193	5.01	5.11	1.0258	6.66	6.83
1.0064	1.67	1.68	1.0129	3.33	3.37	1.0194	5.04	5.14	1.0259	6.69	6.86

\*Calculated from results obtained by drying below 75° C.

TABLE III.—*Extract in beer wort*—Continued.

Extract.			Extract.			Extract.			Extract.		
Specific gravity at 15°C.	Per cent by weight.	Grams per 100 cc.	Specific gravity at 15°C.	Per cent by weight.	Grams per 100 cc.	Specific gravity at 15°C.	Per cent by weight.	Grams per 100 cc.	Specific gravity at 15°C.	Per cent by weight.	Grams per 100 cc.
1.0260	6.71	6.88	1.0325	8.27	8.54	1.0390	9.92	10.31	1.0455	11.53	12.05
1.0261	6.74	6.92	1.0326	8.29	8.56	1.0391	9.95	10.34	1.0456	11.55	12.08
1.0262	6.77	6.95	1.0327	8.32	8.59	1.0392	9.97	10.36	1.0457	11.57	12.10
1.0263	6.80	6.98	1.0328	8.34	8.61	1.0393	9.99	10.38	1.0458	11.60	12.13
1.0264	6.82	7.00	1.0329	8.37	8.65	1.0394	10.02	10.41	1.0459	11.62	12.15
1.0265	6.85	7.03	1.0330	8.40	8.68	1.0395	10.04	10.44	1.0460	11.65	12.19
1.0266	6.88	7.06	1.0331	8.43	8.71	1.0396	10.06	10.46	1.0461	11.67	12.21
1.0267	6.91	7.09	1.0332	8.45	8.73	1.0397	10.09	10.49	1.0462	11.70	12.24
1.0268	6.93	7.12	1.0333	8.48	8.76	1.0398	10.11	10.51	1.0463	11.72	12.26
1.0269	6.96	7.15	1.0334	8.51	8.79	1.0399	10.13	10.53	1.0464	11.75	12.30
1.0270	6.99	7.18	1.0335	8.53	8.82	1.0400	10.16	10.57	1.0465	11.77	12.32
1.0271	7.01	7.20	1.0336	8.56	8.85	1.0401	10.18	10.59	1.0466	11.79	12.34
1.0272	7.04	7.23	1.0337	8.59	8.88	1.0402	10.20	10.61	1.0467	11.82	12.37
1.0273	7.07	7.26	1.0338	8.61	8.90	1.0403	10.23	10.64	1.0468	11.84	12.39
1.0274	7.10	7.29	1.0339	8.64	8.93	1.0404	10.25	10.66	1.0469	11.87	12.43
1.0275	7.12	7.32	1.0340	8.67	8.96	1.0405	10.27	10.69	1.0470	11.89	12.45
1.0276	7.15	7.35	1.0341	8.70	9.00	1.0406	10.30	10.72	1.0471	11.92	12.48
1.0277	7.18	7.38	1.0342	8.72	9.02	1.0407	10.32	10.74	1.0472	11.94	12.50
1.0278	7.21	7.41	1.0343	8.75	9.05	1.0408	10.35	10.77	1.0473	11.97	12.54
1.0279	7.23	7.43	1.0344	8.78	9.08	1.0409	10.37	10.79	1.0474	11.99	12.56
1.0280	7.26	7.46	1.0345	8.80	9.10	1.0410	10.40	10.83	1.0475	12.01	12.58
1.0281	7.28	7.48	1.0346	8.83	9.14	1.0411	10.42	10.85	1.0476	12.04	12.61
1.0282	7.30	7.51	1.0347	8.86	9.17	1.0412	10.45	10.88	1.0477	12.06	12.64
1.0283	7.33	7.54	1.0348	8.88	9.19	1.0413	10.47	10.90	1.0478	12.09	12.67
1.0284	7.35	7.56	1.0349	8.91	9.22	1.0414	10.50	10.93	1.0479	12.11	12.69
1.0285	7.37	7.58	1.0350	8.94	9.25	1.0415	10.52	10.96	1.0480	12.14	12.72
1.0286	7.39	7.60	1.0351	8.97	9.28	1.0416	10.55	10.99	1.0481	12.16	12.74
1.0287	7.42	7.63	1.0352	8.99	9.31	1.0417	10.57	11.01	1.0482	12.19	12.78
1.0288	7.44	7.65	1.0353	9.02	9.34	1.0418	10.60	11.04	1.0483	12.21	12.80
1.0289	7.46	7.68	1.0354	9.05	9.37	1.0419	10.62	11.06	1.0484	12.23	12.82
1.0290	7.48	7.70	1.0355	9.07	9.39	1.0420	10.65	11.10	1.0485	12.26	12.85
1.0291	7.51	7.73	1.0356	9.10	9.42	1.0421	10.67	11.12	1.0486	12.28	12.88
1.0292	7.53	7.75	1.0357	9.13	9.46	1.0422	10.70	11.15	1.0487	12.31	12.91
1.0293	7.55	7.77	1.0358	9.15	9.48	1.0423	10.72	11.17	1.0488	12.33	12.93
1.0294	7.57	7.79	1.0359	9.18	9.51	1.0424	10.75	11.21	1.0489	12.36	12.96
1.0295	7.60	7.82	1.0360	9.21	9.54	1.0425	10.77	11.23	1.0490	12.38	12.99
1.0296	7.62	7.85	1.0361	9.24	9.57	1.0426	10.80	11.26	1.0491	12.41	13.02
1.0297	7.64	7.87	1.0362	9.26	9.60	1.0427	10.82	11.28	1.0492	12.43	13.04
1.0298	7.66	7.89	1.0363	9.29	9.63	1.0428	10.85	11.31	1.0493	12.45	13.06
1.0299	7.69	7.92	1.0364	9.31	9.65	1.0429	10.88	11.35	1.0494	12.48	13.10
1.0300	7.71	7.94	1.0365	9.34	9.68	1.0430	10.90	11.37	1.0495	12.50	13.12
1.0301	7.73	7.96	1.0366	9.36	9.70	1.0431	10.93	11.40	1.0496	12.53	13.15
1.0302	7.75	7.98	1.0367	9.38	9.72	1.0432	10.95	11.42	1.0497	12.55	13.17
1.0303	7.77	8.01	1.0368	9.41	9.76	1.0433	10.98	11.46	1.0498	12.58	13.21
1.0304	7.80	8.04	1.0369	9.43	9.78	1.0434	11.00	11.48	1.0499	12.60	13.23
1.0305	7.82	8.06	1.0370	9.45	9.80	1.0435	11.03	11.51	1.0500	12.63	13.26
1.0306	7.84	8.08	1.0371	9.48	9.83	1.0436	11.05	11.53	1.0501	12.65	13.28
1.0307	7.86	8.10	1.0372	9.50	9.85	1.0437	11.08	11.56	1.0502	12.67	13.31
1.0308	7.89	8.13	1.0373	9.52	9.88	1.0438	11.10	11.59	1.0503	12.70	13.34
1.0309	7.91	8.15	1.0374	9.55	9.91	1.0439	11.13	11.62	1.0504	12.72	13.36
1.0310	7.93	8.18	1.0375	9.57	9.93	1.0440	11.15	11.64	1.0505	12.75	13.39
1.0311	7.95	8.20	1.0376	9.59	9.95	1.0441	11.18	11.67	1.0506	12.77	13.42
1.0312	7.98	8.23	1.0377	9.62	9.98	1.0442	11.20	11.70	1.0507	12.80	13.45
1.0313	8.00	8.25	1.0378	9.64	10.00	1.0443	11.23	11.73	1.0508	12.82	13.47
1.0314	8.02	8.27	1.0379	9.66	10.03	1.0444	11.25	11.75	1.0509	12.85	13.50
1.0315	8.04	8.29	1.0380	9.69	10.06	1.0445	11.28	11.78	1.0510	12.87	13.53
1.0316	8.07	8.33	1.0381	9.71	10.08	1.0446	11.30	11.80	1.0511	12.90	13.56
1.0317	8.09	8.35	1.0382	9.73	10.10	1.0447	11.33	11.84	1.0512	12.92	13.58
1.0318	8.11	8.37	1.0383	9.76	10.13	1.0448	11.35	11.86	1.0513	12.94	13.60
1.0319	8.13	8.39	1.0384	9.78	10.16	1.0449	11.38	11.89	1.0514	12.97	13.64
1.0320	8.16	8.42	1.0385	9.81	10.19	1.0450	11.40	11.91	1.0515	12.99	13.66
1.0321	8.18	8.44	1.0386	9.83	10.21	1.0451	11.43	11.95	1.0516	12.02	13.69
1.0322	8.20	8.46	1.0387	9.85	10.23	1.0452	11.45	11.97	1.0517	12.04	13.71
1.0323	8.22	8.49	1.0388	9.88	10.26	1.0453	11.48	12.00	1.0518	12.07	13.75
1.0324	8.25	8.52	1.0389	9.90	10.29	1.0454	11.50	12.02	1.0519	12.09	13.77

TABLE III.—*Extract in beer wort*—Continued.

Specific gravity at 15° C.	Extract.		Specific gravity at 15° C.	Extract.		Specific gravity at 15° C.	Extract.		Specific gravity at 15° C.	Extract.	
	Per cent by weight.	Grams per 100 cc.		Per cent by weight.	Grams per 100 cc.		Per cent by weight.	Grams per 100 cc.		Per cent by weight.	Grams per 100 cc.
1.0520	13.12	13.80	1.0585	14.75	15.61	1.0650	16.25	17.31	1.0715	17.81	19.08
1.0521	13.14	13.82	1.0586	14.78	15.65	1.0651	16.27	17.33	1.0716	17.84	19.12
1.0522	13.16	13.85	1.0587	14.81	15.68	1.0652	16.30	17.36	1.0717	17.86	19.14
1.0523	13.19	13.88	1.0588	14.83	15.70	1.0653	16.32	17.39	1.0718	17.88	19.16
1.0524	13.21	13.90	1.0589	14.86	15.74	1.0654	16.35	17.42	1.0719	17.90	19.19
1.0525	13.24	13.94	1.0590	14.89	15.77	1.0655	16.37	17.44	1.0720	17.93	19.22
1.0526	13.26	13.96	1.0591	14.91	15.79	1.0656	16.40	17.48	1.0721	17.95	19.24
1.0527	13.29	13.99	1.0592	14.94	15.82	1.0657	16.42	17.50	1.0722	17.97	19.27
1.0528	13.31	14.01	1.0593	14.96	15.85	1.0658	16.45	17.53	1.0723	17.99	19.29
1.0529	13.34	14.05	1.0594	14.99	15.88	1.0659	16.47	17.56	1.0724	18.02	19.32
1.0530	13.36	14.07	1.0595	15.02	15.91	1.0660	16.50	17.59	1.0725	18.04	19.35
1.0531	13.38	14.09	1.0596	15.04	15.94	1.0661	16.52	17.61	1.0726	18.06	19.37
1.0532	13.41	14.12	1.0597	15.07	15.97	1.0662	16.54	17.63	1.0727	18.08	19.39
1.0533	13.43	14.15	1.0598	15.09	15.99	1.0663	16.57	17.67	1.0728	18.11	19.43
1.0534	13.46	14.18	1.0599	15.11	16.02	1.0664	16.59	17.69	1.0729	18.13	19.45
1.0535	13.48	14.20	1.0600	15.14	16.05	1.0665	16.62	17.73	1.0730	18.15	19.47
1.0536	13.51	14.23	1.0601	15.16	16.07	1.0666	16.64	17.75	1.0731	18.17	19.50
1.0537	13.53	14.26	1.0602	15.18	16.09	1.0667	16.67	17.78	1.0732	18.20	19.53
1.0538	13.56	14.29	1.0603	15.20	16.12	1.0668	16.69	17.80	1.0733	18.22	19.55
1.0539	13.58	14.31	1.0604	15.23	16.15	1.0669	16.72	17.84	1.0734	18.24	19.58
1.0540	13.61	14.34	1.0605	15.25	16.17	1.0670	16.74	17.86	1.0735	18.26	19.60
1.0541	13.63	14.37	1.0606	15.27	16.20	1.0671	16.76	17.88	1.0736	18.29	19.64
1.0542	13.66	14.40	1.0607	15.29	16.22	1.0672	16.79	17.92	1.0737	18.31	19.66
1.0543	13.68	14.42	1.0608	15.31	16.24	1.0673	16.81	17.94	1.0738	18.33	19.68
1.0544	13.71	14.46	1.0609	15.34	16.27	1.0674	16.84	17.98	1.0739	18.35	19.71
1.0545	13.73	14.48	1.0610	15.36	16.30	1.0675	16.86	18.00	1.0740	18.38	19.74
1.0546	13.76	14.51	1.0611	15.38	16.32	1.0676	16.89	18.03	1.0741	18.40	19.76
1.0547	13.78	14.53	1.0612	15.40	16.34	1.0677	16.91	18.05	1.0742	18.42	19.79
1.0548	13.81	14.57	1.0613	15.43	16.38	1.0678	16.94	18.09	1.0743	18.44	19.81
1.0549	13.83	14.59	1.0614	15.45	16.40	1.0679	16.96	18.11	1.0744	18.47	19.84
1.0550	13.86	14.62	1.0615	15.47	16.42	1.0680	16.99	18.15	1.0745	18.49	19.87
1.0551	13.88	14.64	1.0616	15.49	16.44	1.0681	17.01	18.17	1.0746	18.51	19.89
1.0552	13.91	14.68	1.0617	15.52	16.48	1.0682	17.03	18.19	1.0747	18.53	19.91
1.0553	13.93	14.70	1.0618	15.54	16.50	1.0683	17.06	18.23	1.0748	18.55	19.94
1.0554	13.96	14.73	1.0619	15.56	16.52	1.0684	17.08	18.25	1.0749	18.57	19.96
1.0555	13.98	14.76	1.0620	15.58	16.55	1.0685	17.11	18.28	1.0750	18.59	19.98
1.0556	14.01	14.79	1.0621	15.60	16.57	1.0686	17.13	18.31	1.0751	18.62	20.02
1.0557	14.03	14.81	1.0622	15.63	16.60	1.0687	17.16	18.34	1.0752	18.64	20.04
1.0558	14.06	14.84	1.0623	15.65	16.62	1.0688	17.18	18.36	1.0753	18.66	20.07
1.0559	14.08	14.87	1.0624	15.67	16.64	1.0689	17.21	18.40	1.0754	18.68	20.09
1.0560	14.11	14.90	1.0625	15.69	16.66	1.0690	17.23	18.42	1.0755	18.70	20.11
1.0561	14.13	14.92	1.0626	15.72	16.70	1.0691	17.25	18.44	1.0756	18.72	20.14
1.0562	14.16	14.96	1.0627	15.74	16.73	1.0692	17.28	18.48	1.0757	18.74	20.16
1.0563	14.18	14.98	1.0628	15.76	16.75	1.0693	17.30	18.50	1.0758	18.76	20.18
1.0564	14.21	15.01	1.0629	15.78	16.77	1.0694	17.33	18.53	1.0759	18.78	20.21
1.0565	14.23	15.03	1.0630	15.80	16.80	1.0695	17.35	18.56	1.0760	18.81	20.24
1.0566	14.26	15.07	1.0631	15.83	16.83	1.0696	17.38	18.59	1.0761	18.83	20.26
1.0567	14.28	15.09	1.0632	15.85	16.85	1.0697	17.40	18.61	1.0762	18.85	20.29
1.0568	14.31	15.12	1.0633	15.87	16.87	1.0698	17.43	18.65	1.0763	18.87	20.31
1.0569	14.33	15.15	1.0634	15.89	16.90	1.0699	17.45	18.67	1.0764	18.89	20.33
1.0570	14.36	15.18	1.0635	15.92	16.93	1.0700	17.48	18.70	1.0765	18.91	20.36
1.0571	14.38	15.20	1.0636	15.94	16.95	1.0701	17.50	18.73	1.0766	18.93	20.38
1.0572	14.41	15.23	1.0637	15.96	16.98	1.0702	17.52	18.75	1.0767	18.95	20.40
1.0573	14.44	15.27	1.0638	15.98	17.00	1.0703	17.54	18.77	1.0768	18.97	20.43
1.0574	14.46	15.29	1.0639	16.01	17.03	1.0704	17.57	18.81	1.0769	19.00	20.46
1.0575	14.49	15.32	1.0640	16.03	17.06	1.0705	17.59	18.83	1.0770	19.02	20.48
1.0576	14.52	15.36	1.0641	16.05	17.08	1.0706	17.61	18.85	1.0771	19.04	20.51
1.0577	14.54	15.38	1.0642	16.07	17.10	1.0707	17.63	18.88	1.0772	19.06	20.53
1.0578	14.57	15.41	1.0643	16.09	17.12	1.0708	17.66	18.91	1.0773	19.08	20.55
1.0579	14.59	15.43	1.0644	16.12	17.16	1.0709	17.68	18.93	1.0774	19.10	20.58
1.0580	14.62	15.47	1.0645	16.14	17.18	1.0710	17.70	18.96	1.0775	19.12	20.60
1.0581	14.65	15.50	1.0646	16.16	17.20	1.0711	17.72	18.98	1.0776	19.14	20.63
1.0582	14.67	15.52	1.0647	16.18	17.23	1.0712	17.75	19.01	1.0777	19.17	20.66
1.0583	14.70	15.56	1.0648	16.21	17.26	1.0713	17.77	19.04	1.0778	19.19	20.68
1.0584	14.73	15.59	1.0649	16.23	17.28	1.0714	17.79	19.06	1.0779	19.21	20.71

TABLE III.—*Extract in beer wort*—Continued.

Specific gravity at 15°C.	Extract.		Specific gravity at 15°C.	Extract.		Specific gravity at 15°C.	Extract.		Specific gravity at 15°C.	Extract.	
	Per cent by weight.	Grams per 100 cc.		Per cent by weight.	Grams per 100 cc.		Per cent by weight.	Grams per 100 cc.		Per cent by weight.	Grams per 100 cc.
1.0780	19.23	20.73	1.0845	20.70	22.45	1.0910	22.19	24.21	1.0975	23.59	25.89
1.0781	19.25	20.75	1.0846	20.73	22.48	1.0911	22.21	24.24	1.0976	23.61	25.92
1.0782	19.27	20.78	1.0847	20.75	22.50	1.0912	22.23	24.26	1.0977	23.63	25.94
1.0783	19.29	20.80	1.0848	20.77	22.53	1.0913	22.26	24.29	1.0978	23.65	25.97
1.0784	19.31	20.82	1.0849	20.79	22.55	1.0914	22.28	24.31	1.0979	23.67	25.99
1.0785	19.33	20.85	1.0850	20.81	22.58	1.0915	22.30	24.34	1.0980	23.69	26.01
1.0786	19.36	20.88	1.0851	20.83	22.61	1.0916	22.32	24.37	1.0981	23.71	26.04
1.0787	19.38	20.90	1.0852	20.86	22.64	1.0917	22.34	24.39	1.0982	23.73	26.06
1.0788	19.40	20.93	1.0853	20.88	22.66	1.0918	22.37	24.42	1.0983	23.76	26.09
1.0789	19.42	20.95	1.0854	20.90	22.68	1.0919	22.39	24.44	1.0984	23.78	26.11
1.0790	19.44	20.98	1.0855	20.93	22.72	1.0920	22.41	24.47	1.0985	23.80	26.14
1.0791	19.46	21.00	1.0856	20.95	22.75	1.0921	22.43	24.49	1.0986	23.82	26.17
1.0792	19.49	21.03	1.0857	20.98	22.78	1.0922	22.45	24.51	1.0987	23.84	26.19
1.0793	19.51	21.06	1.0858	21.01	22.81	1.0923	22.48	24.54	1.0988	23.86	26.22
1.0794	19.53	21.08	1.0859	21.04	22.84	1.0924	22.50	24.56	1.0989	23.88	26.24
1.0795	19.56	21.11	1.0860	21.06	22.87	1.0925	22.52	24.60	1.0990	23.90	26.27
1.0796	19.58	21.14	1.0861	21.09	22.90	1.0926	22.54	24.62	1.0991	23.92	26.30
1.0797	19.60	21.16	1.0862	21.11	22.93	1.0927	22.56	24.64	1.0992	23.94	26.32
1.0798	19.63	21.20	1.0863	21.13	22.96	1.0928	22.59	24.67	1.0993	23.97	26.35
1.0799	19.65	21.22	1.0864	21.16	22.99	1.0929	22.61	24.70	1.0994	23.99	26.37
1.0800	19.67	21.24	1.0865	21.19	23.02	1.0930	22.63	24.73	1.0995	24.01	26.40
1.0801	19.70	21.28	1.0866	21.22	23.06	1.0931	22.65	24.76	1.0996	24.03	26.42
1.0802	19.72	21.30	1.0867	21.25	23.09	1.0932	22.67	24.78	1.0997	24.05	26.44
1.0803	19.74	21.33	1.0868	21.28	23.12	1.0933	22.69	24.81	1.0998	24.07	26.47
1.0804	19.77	21.36	1.0869	21.30	23.15	1.0934	22.71	24.83	1.0999	24.09	26.49
1.0805	19.79	21.38	1.0870	21.33	23.18	1.0935	22.73	24.86	1.1000	24.11	26.52
1.0806	19.81	21.41	1.0871	21.35	23.21	1.0936	22.75	24.89	1.1001	24.13	26.55
1.0807	19.84	21.43	1.0872	21.37	23.23	1.0937	22.77	24.91	1.1002	24.15	26.57
1.0808	19.86	21.46	1.0873	21.39	23.26	1.0938	22.80	24.93	1.1003	24.17	26.60
1.0809	19.88	21.49	1.0874	21.41	23.28	1.0939	22.82	24.96	1.1004	24.19	26.62
1.0810	19.91	21.52	1.0875	21.43	23.31	1.0940	22.84	24.99	1.1005	24.21	26.65
1.0811	19.93	21.55	1.0876	21.45	23.33	1.0941	22.86	25.01	1.1006	24.23	26.68
1.0812	19.96	21.58	1.0877	21.47	23.36	1.0942	22.88	25.03	1.1007	24.25	26.70
1.0813	19.98	21.60	1.0878	21.49	23.38	1.0943	22.90	25.06	1.1008	24.28	26.73
1.0814	20.00	21.63	1.0879	21.51	23.40	1.0944	22.92	25.08	1.1009	24.30	26.75
1.0815	20.03	21.66	1.0880	21.54	23.43	1.0945	22.94	25.11	1.1010	24.32	26.78
1.0816	20.05	21.69	1.0881	21.56	23.45	1.0946	22.96	25.14	1.1011	24.34	26.81
1.0817	20.07	21.71	1.0882	21.58	23.48	1.0947	22.98	25.16	1.1012	24.36	26.83
1.0818	20.10	21.74	1.0883	21.60	23.50	1.0948	23.00	25.18	1.1013	24.39	26.86
1.0819	20.12	21.77	1.0884	21.62	23.52	1.0949	23.03	25.21	1.1014	24.41	26.88
1.0820	20.14	21.79	1.0885	21.64	23.55	1.0950	23.05	25.24	1.1015	24.43	26.91
1.0821	20.17	21.83	1.0886	21.66	23.58	1.0951	23.07	25.26	1.1016	24.45	26.93
1.0822	20.19	21.85	1.0887	21.68	23.60	1.0952	23.10	25.29	1.1017	24.47	26.95
1.0823	20.21	21.87	1.0888	21.71	23.63	1.0953	23.12	25.31	1.1018	24.49	26.98
1.0824	20.24	21.91	1.0889	21.73	23.66	1.0954	23.14	25.34	1.1019	24.51	27.00
1.0825	20.26	21.93	1.0890	21.75	23.69	1.0955	23.16	25.37	1.1020	24.53	27.03
1.0826	20.28	21.96	1.0891	21.77	23.72	1.0956	23.18	25.39	1.1021	24.55	27.06
1.0827	20.31	21.99	1.0892	21.79	23.74	1.0957	23.20	25.42	1.1022	24.57	27.08
1.0828	20.33	22.01	1.0893	21.82	23.77	1.0958	23.23	25.45	1.1023	24.60	27.11
1.0829	20.35	22.04	1.0894	21.84	23.79	1.0959	23.25	25.47	1.1024	24.62	27.14
1.0830	20.37	22.06	1.0895	21.86	23.82	1.0960	23.27	25.50	1.1025	24.64	27.17
1.0831	20.39	22.08	1.0896	21.89	23.85	1.0961	23.29	25.53	1.1026	24.66	27.19
1.0832	20.41	22.11	1.0897	21.91	23.87	1.0962	23.31	25.55	1.1027	24.68	27.21
1.0833	20.43	22.13	1.0898	21.93	23.90	1.0963	23.33	25.58	1.1028	24.70	27.24
1.0834	20.46	22.16	1.0899	21.96	23.93	1.0964	23.35	25.60	1.1029	24.72	27.26
1.0835	20.48	22.19	1.0900	21.98	23.96	1.0965	23.37	25.63	1.1030	24.74	27.29
1.0836	20.50	22.21	1.0901	22.00	23.98	1.0966	23.39	25.66	1.1031	24.76	27.32
1.0837	20.52	22.24	1.0902	22.02	24.01	1.0967	23.41	25.68	1.1032	24.78	27.34
1.0838	20.54	22.26	1.0903	22.04	24.03	1.0968	23.44	25.71	1.1033	24.81	27.37
1.0839	20.56	22.29	1.0904	22.06	24.05	1.0969	23.46	25.73	1.1034	24.83	27.39
1.0840	20.59	22.32	1.0905	22.08	24.08	1.0970	23.48	25.76	1.1035	24.85	27.42
1.0841	20.62	22.35	1.0906	22.10	24.11	1.0971	23.50	25.79	1.1036	24.87	27.45
1.0842	20.64	22.38	1.0907	22.12	24.13	1.0972	23.52	25.81	1.1037	24.89	27.47
1.0843	20.66	22.40	1.0908	22.15	24.16	1.0973	23.55	25.84	1.1038	24.92	27.50
1.0844	20.68	22.42	1.0909	22.17	24.18	1.0974	23.57	25.86	1.1039	24.94	27.53

TABLE III.—*Extract in beer wort*—Continued.

Specific gravity at 15° C.	Extract.		Specific gravity at 15° C.	Extract.		Specific gravity at 15° C.	Extract.		Specific gravity at 15° C.	Extract.	
	Per cent by weight.	Grams per 100 cc.		Per cent by weight.	Grams per 100 cc.		Per cent by weight.	Grams per 100 cc.		Per cent by weight.	Grams per 100 cc.
1.1040	24.96	27.56	1.1095	26.16	29.03	1.1150	27.29	30.43	1.1205	28.38	31.81
1.1041	24.98	27.58	1.1096	26.18	29.06	1.1151	27.31	30.45	1.1206	28.40	31.83
1.1042	25.00	27.60	1.1097	26.20	29.08	1.1152	27.33	30.47	1.1207	28.42	31.86
1.1043	25.03	27.63	1.1098	26.23	29.11	1.1153	27.35	30.50	1.1208	28.44	31.88
1.1044	25.05	27.66	1.1099	26.25	29.13	1.1154	27.37	30.52	1.1209	28.46	31.90
1.1045	25.07	27.69	1.1100	26.27	29.16	1.1155	27.38	30.55	1.1210	28.48	31.93
1.1046	25.09	27.72	1.1101	26.29	29.19	1.1156	27.40	30.57	1.1211	28.50	31.95
1.1047	25.11	27.74	1.1102	26.31	29.21	1.1157	27.42	30.59	1.1212	28.52	31.98
1.1048	25.14	27.77	1.1103	26.33	29.24	1.1158	27.44	30.62	1.1213	28.54	32.00
1.1049	25.16	27.79	1.1104	26.35	29.26	1.1159	27.46	30.64	1.1214	28.56	32.03
1.1050	25.18	27.82	1.1105	26.37	29.29	1.1160	27.48	30.67	1.1215	28.58	32.05
1.1051	25.20	27.85	1.1106	26.39	29.32	1.1161	27.50	30.69	1.1216	28.60	32.08
1.1052	25.22	27.87	1.1107	26.41	29.34	1.1162	27.52	30.72	1.1217	28.62	32.11
1.1053	25.24	27.90	1.1108	26.44	29.37	1.1163	27.54	30.75	1.1218	28.64	32.13
1.1054	25.27	27.93	1.1109	26.46	29.39	1.1164	27.56	30.77	1.1219	28.66	32.15
1.1055	25.29	27.96	1.1110	26.48	29.42	1.1165	27.58	30.80	1.1220	28.68	32.18
1.1056	25.31	27.98	1.1111	26.50	29.44	1.1166	27.60	30.82	1.1221	28.70	32.20
1.1057	25.33	28.00	1.1112	26.52	29.46	1.1167	27.62	30.85	1.1222	28.72	32.23
1.1058	25.35	28.03	1.1113	26.54	29.49	1.1168	27.64	30.87	1.1223	28.74	32.25
1.1059	25.38	28.06	1.1114	26.56	29.51	1.1169	27.66	30.89	1.1224	28.76	32.27
1.1060	25.40	28.09	1.1115	26.58	29.54	1.1170	27.68	30.92	1.1225	28.78	32.30
1.1061	25.42	28.12	1.1116	26.60	29.57	1.1171	27.70	30.94	1.1226	28.80	32.32
1.1062	25.44	28.14	1.1117	26.62	29.59	1.1172	27.72	30.97	1.1227	28.82	32.35
1.1063	25.46	28.17	1.1118	26.64	29.61	1.1173	27.74	31.00	1.1228	28.84	32.37
1.1064	25.48	28.19	1.1119	26.66	29.64	1.1174	27.76	31.02	1.1229	28.86	32.40
1.1065	25.50	28.22	1.1120	26.68	29.67	1.1175	27.78	31.05	1.1230	28.88	32.43
1.1066	25.52	28.25	1.1121	26.70	29.69	1.1176	27.80	31.07	1.1231	28.90	32.45
1.1067	25.54	28.27	1.1122	26.72	29.71	1.1177	27.82	31.09	1.1232	28.92	32.48
1.1068	25.57	28.30	1.1123	26.75	29.74	1.1178	27.84	31.12	1.1233	28.94	32.50
1.1069	25.59	28.32	1.1124	26.77	29.77	1.1179	27.86	31.15	1.1234	28.96	32.53
1.1070	25.61	28.35	1.1125	26.79	29.80	1.1180	27.88	31.18	1.1235	28.98	32.56
1.1071	25.63	28.38	1.1126	26.81	29.83	1.1181	27.90	31.20	1.1236	29.00	32.58
1.1072	25.65	28.40	1.1127	26.83	29.85	1.1182	27.92	31.23	1.1237	29.02	32.60
1.1073	25.67	28.43	1.1128	26.85	29.88	1.1183	27.94	31.25	1.1238	29.04	32.63
1.1074	25.69	28.45	1.1129	26.87	29.90	1.1184	27.96	31.27	1.1239	29.06	32.65
1.1075	25.71	28.48	1.1130	26.89	29.93	1.1185	27.98	31.30	1.1240	29.08	32.68
1.1076	25.73	28.51	1.1131	26.91	29.95	1.1186	28.00	31.32	1.1241	29.10	32.71
1.1077	25.75	28.53	1.1132	26.93	29.97	1.1187	28.02	31.35	1.1242	29.12	32.73
1.1078	25.78	28.56	1.1133	26.95	30.00	1.1188	28.04	31.37	1.1243	29.14	32.76
1.1079	25.80	28.58	1.1134	26.97	30.02	1.1189	28.07	31.40	1.1244	29.16	32.78
1.1080	25.82	28.61	1.1135	26.99	30.06	1.1190	28.09	31.43	1.1245	29.18	32.81
1.1081	25.84	28.64	1.1136	27.01	30.08	1.1191	28.11	31.45	1.1246	29.20	32.83
1.1082	25.86	28.66	1.1137	27.03	30.10	1.1192	28.13	31.48	1.1247	29.22	32.86
1.1083	25.89	28.69	1.1138	27.05	30.13	1.1193	28.15	31.51	1.1248	29.24	32.89
1.1084	25.91	28.72	1.1139	27.07	30.15	1.1194	28.17	31.53	1.1249	29.26	32.91
1.1085	25.93	28.75	1.1140	27.09	30.18	1.1195	28.19	31.56	1.1250	29.28	32.94
1.1086	25.96	28.78	1.1141	27.11	30.20	1.1196	28.21	31.59	1.1251	29.30	32.96
1.1087	25.98	28.80	1.1142	27.13	30.22	1.1197	28.23	31.61	1.1252	29.32	32.99
1.1088	26.01	28.83	1.1143	27.15	30.25	1.1198	28.25	31.63	1.1253	29.34	33.02
1.1089	26.03	28.86	1.1144	27.17	30.27	1.1199	28.27	31.65	1.1254	29.36	33.04
1.1090	26.05	28.89	1.1145	27.19	30.31	1.1200	28.28	31.68	1.1255	29.38	33.07
1.1091	26.07	28.92	1.1146	27.21	30.33	1.1201	28.30	31.70	1.1256	29.40	33.09
1.1092	26.09	28.94	1.1147	27.23	30.35	1.1202	28.32	31.73	1.1257	29.42	33.12
1.1093	26.12	28.97	1.1148	27.25	30.37	1.1203	28.34	31.75	1.1258	29.44	33.14
1.1094	26.14	29.00	1.1149	27.27	30.40	1.1204	28.36	31.78	1.1259	29.47	33.17

TABLE IV.—*Extract in beer wort.\**

[According to H. Ellion.]

Specific gravity at 15° C.	Extract.		Specific gravity at 15° C.	Extract.		Specific gravity at 15° C.	Extract.		Specific gravity at 15° C.	Extract.	
	Per cent by weight.	Grams per 100 cc.		Per cent by weight.	Grams per 100 cc.		Per cent by weight.	Grams per 100 cc.		Per cent by weight.	Grams per 100 cc.
1.0001	0.02	0.02	1.0066	1.63	1.64	1.0131	3.22	3.26	1.0196	4.79	4.89
1.0002	0.05	0.05	1.0067	1.66	1.67	1.0132	3.25	3.29	1.0197	4.82	4.91
1.0003	0.07	0.07	1.0068	1.68	1.69	1.0133	3.27	3.31	1.0198	4.84	4.94
1.0004	0.10	0.10	1.0069	1.71	1.72	1.0134	3.29	3.34	1.0199	4.87	4.96
1.0005	0.12	0.12	1.0070	1.73	1.74	1.0135	3.32	3.36	1.0200	4.89	4.99
1.0006	0.15	0.15	1.0071	1.76	1.77	1.0136	3.34	3.39	1.0201	4.91	5.01
1.0007	0.17	0.17	1.0072	1.78	1.79	1.0137	3.37	3.41	1.0202	4.94	5.04
1.0008	0.20	0.20	1.0073	1.80	1.82	1.0138	3.39	3.44	1.0203	4.96	5.06
1.0009	0.22	0.22	1.0074	1.83	1.84	1.0139	3.42	3.46	1.0204	4.99	5.09
1.0010	0.25	0.25	1.0075	1.85	1.87	1.0140	3.44	3.49	1.0205	5.01	5.11
1.0011	0.27	0.27	1.0076	1.88	1.89	1.0141	3.46	3.51	1.0206	5.03	5.14
1.0012	0.30	0.30	1.0077	1.90	1.92	1.0142	3.49	3.54	1.0207	5.06	5.16
1.0013	0.32	0.32	1.0078	1.93	1.94	1.0143	3.51	3.56	1.0208	5.08	5.19
1.0014	0.35	0.35	1.0079	1.95	1.97	1.0144	3.54	3.59	1.0209	5.11	5.21
1.0015	0.37	0.37	1.0080	1.98	1.99	1.0145	3.56	3.61	1.0210	5.13	5.24
1.0016	0.40	0.40	1.0081	2.00	2.02	1.0146	3.59	3.64	1.0211	5.15	5.26
1.0017	0.42	0.42	1.0082	2.03	2.04	1.0147	3.61	3.66	1.0212	5.18	5.29
1.0018	0.45	0.45	1.0083	2.05	2.07	1.0148	3.63	3.69	1.0213	5.20	5.31
1.0019	0.47	0.47	1.0084	2.07	2.09	1.0149	3.66	3.71	1.0214	5.23	5.34
1.0020	0.50	0.50	1.0085	2.10	2.12	1.0150	3.68	3.74	1.0215	5.25	5.36
1.0021	0.52	0.52	1.0086	2.12	2.14	1.0151	3.71	3.76	1.0216	5.27	5.39
1.0022	0.55	0.55	1.0087	2.15	2.17	1.0152	3.73	3.79	1.0217	5.30	5.41
1.0023	0.57	0.57	1.0088	2.17	2.19	1.0153	3.76	3.81	1.0218	5.32	5.44
1.0024	0.60	0.60	1.0089	2.20	2.22	1.0154	3.78	3.84	1.0219	5.35	5.46
1.0025	0.62	0.62	1.0090	2.22	2.24	1.0155	3.80	3.86	1.0220	5.37	5.49
1.0026	0.65	0.65	1.0091	2.25	2.27	1.0156	3.83	3.89	1.0221	5.39	5.51
1.0027	0.67	0.67	1.0092	2.27	2.29	1.0157	3.85	3.91	1.0222	5.42	5.54
1.0028	0.69	0.70	1.0093	2.29	2.32	1.0158	3.88	3.94	1.0223	5.44	5.56
1.0029	0.72	0.72	1.0094	2.32	2.34	1.0159	3.90	3.96	1.0224	5.47	5.59
1.0030	0.74	0.75	1.0095	2.34	2.37	1.0160	3.93	3.99	1.0225	5.49	5.61
1.0031	0.77	0.77	1.0096	2.37	2.39	1.0161	3.95	4.01	1.0226	5.51	5.64
1.0032	0.79	0.80	1.0097	2.39	2.42	1.0162	3.97	4.04	1.0227	5.54	5.66
1.0033	0.82	0.82	1.0098	2.42	2.44	1.0163	4.00	4.06	1.0228	5.56	5.69
1.0034	0.84	0.85	1.0099	2.44	2.47	1.0164	4.02	4.09	1.0229	5.59	5.71
1.0035	0.87	0.87	1.0100	2.47	2.49	1.0165	4.05	4.11	1.0230	5.61	5.74
1.0036	0.90	0.90	1.0101	2.49	2.52	1.0166	4.07	4.14	1.0231	5.63	5.76
1.0037	0.92	0.92	1.0102	2.51	2.54	1.0167	4.09	4.16	1.0232	5.66	5.79
1.0038	0.94	0.95	1.0103	2.54	2.57	1.0168	4.12	4.19	1.0233	5.68	5.81
1.0039	0.97	0.97	1.0104	2.56	2.59	1.0169	4.14	4.21	1.0234	5.70	5.84
1.0040	0.99	1.00	1.0105	2.59	2.62	1.0170	4.17	4.24	1.0235	5.73	5.86
1.0041	1.02	1.02	1.0106	2.61	2.64	1.0171	4.19	4.26	1.0236	5.75	5.89
1.0042	1.04	1.05	1.0107	2.64	2.67	1.0172	4.22	4.29	1.0237	5.78	5.91
1.0043	1.07	1.07	1.0108	2.66	2.69	1.0173	4.24	4.31	1.0238	5.80	5.94
1.0044	1.09	1.10	1.0109	2.69	2.72	1.0174	4.26	4.34	1.0239	5.82	5.96
1.0045	1.12	1.12	1.0110	2.71	2.74	1.0175	4.29	4.36	1.0240	5.85	5.99
1.0046	1.14	1.14	1.0111	2.73	2.77	1.0176	4.31	4.39	1.0241	5.87	6.01
1.0047	1.16	1.17	1.0112	2.76	2.79	1.0177	4.34	4.41	1.0242	5.90	6.04
1.0048	1.19	1.19	1.0113	2.78	2.81	1.0178	4.36	4.44	1.0243	5.92	6.06
1.0049	1.21	1.22	1.0114	2.81	2.84	1.0179	4.38	4.46	1.0244	5.94	6.09
1.0050	1.24	1.24	1.0115	2.83	2.86	1.0180	4.41	4.49	1.0245	5.97	6.11
1.0051	1.26	1.27	1.0116	2.86	2.89	1.0181	4.43	4.51	1.0246	5.99	6.14
1.0052	1.29	1.29	1.0117	2.88	2.91	1.0182	4.46	4.54	1.0247	6.01	6.16
1.0053	1.31	1.32	1.0118	2.91	2.94	1.0183	4.48	4.56	1.0248	6.04	6.19
1.0054	1.34	1.34	1.0119	2.93	2.96	1.0184	4.50	4.59	1.0249	6.06	6.21
1.0055	1.36	1.37	1.0120	2.95	2.99	1.0185	4.53	4.61	1.0250	6.09	6.24
1.0056	1.39	1.39	1.0121	2.98	3.01	1.0186	4.55	4.64	1.0251	6.11	6.26
1.0057	1.41	1.42	1.0122	3.00	3.04	1.0187	4.58	4.66	1.0252	6.13	6.29
1.0058	1.44	1.44	1.0123	3.03	3.06	1.0188	4.60	4.69	1.0253	6.16	6.31
1.0059	1.46	1.47	1.0124	3.05	3.09	1.0189	4.63	4.71	1.0254	6.18	6.34
1.0060	1.48	1.49	1.0125	3.08	3.11	1.0190	4.65	4.74	1.0255	6.21	6.36
1.0061	1.51	1.52	1.0126	3.10	3.14	1.0191	4.67	4.76	1.0256	6.23	6.39
1.0062	1.53	1.54	1.0127	3.12	3.16	1.0192	4.70	4.79	1.0257	6.25	6.41
1.0063	1.57	1.56	1.0128	3.15	3.19	1.0193	4.72	4.81	1.0258	6.28	6.44
1.0064	1.58	1.59	1.0129	3.17	3.21	1.0194	4.75	4.84	1.0259	6.30	6.46
1.0065	1.61	1.62	1.0130	3.20	3.24	1.0195	4.77	4.86	1.0260	6.32	6.49

\* Calculated from results obtained by drying at 97° C.

TABLE IV.—*Extract in beer wort*—Continued.

Specific gravity at 15° C.	Extract.		Specific gravity at 15° C.	Extract.		Specific gravity at 15° C.	Extract.		Specific gravity at 15° C.	Extract.	
	Per cent by weight.	Grams per 100 cc.		Per cent by weight.	Grams per 100 cc.		Per cent by weight.	Grams per 100 cc.		Per cent by weight.	Grams per 100 cc.
1.0261	6.35	6.51	1.0326	7.89	8.14	1.0391	9.41	9.77	1.0456	10.91	11.41
1.0262	6.37	6.51	1.0327	7.91	8.17	1.0392	9.43	9.80	1.0457	10.93	11.43
1.0263	6.40	6.56	1.0328	7.93	8.19	1.0393	9.45	9.82	1.0458	10.96	11.46
1.0264	6.42	6.59	1.0329	7.96	8.22	1.0394	9.48	9.85	1.0459	10.98	11.48
1.0265	6.44	6.61	1.0330	7.98	8.24	1.0395	9.50	9.87	1.0460	11.00	11.51
1.0266	6.47	6.64	1.0331	8.00	8.27	1.0396	9.52	9.90	1.0461	11.03	11.53
1.0267	6.49	6.66	1.0332	8.03	8.29	1.0397	9.55	9.92	1.0462	11.05	11.56
1.0268	6.51	6.69	1.0333	8.05	8.32	1.0398	9.57	9.95	1.0463	11.07	11.58
1.0269	6.51	6.71	1.0334	8.07	8.34	1.0399	9.59	9.97	1.0464	11.09	11.61
1.0270	6.56	6.74	1.0335	8.10	8.37	1.0400	9.62	10.00	1.0465	11.12	11.63
1.0271	6.59	6.76	1.0336	8.12	8.39	1.0401	9.64	10.03	1.0466	11.14	11.66
1.0272	6.61	6.79	1.0337	8.14	8.42	1.0402	9.66	10.05	1.0467	11.16	11.68
1.0273	6.63	6.81	1.0338	8.17	8.44	1.0403	9.69	10.08	1.0468	11.19	11.71
1.0274	6.66	6.84	1.0339	8.19	8.47	1.0404	9.71	10.10	1.0469	11.21	11.74
1.0275	6.68	6.86	1.0340	8.21	8.49	1.0405	9.73	10.13	1.0470	11.23	11.76
1.0276	6.70	6.89	1.0341	8.24	8.52	1.0406	9.75	10.15	1.0471	11.26	11.79
1.0277	6.73	6.91	1.0342	8.56	8.54	1.0407	9.78	10.18	1.0472	11.28	11.81
1.0278	6.75	6.94	1.0343	8.28	8.57	1.0408	9.80	10.20	1.0473	11.30	11.84
1.0279	6.78	6.96	1.0344	8.31	8.59	1.0409	9.82	10.23	1.0474	11.32	11.86
1.0280	6.80	6.99	1.0345	8.33	8.62	1.0410	9.85	10.25	1.0475	11.35	11.89
1.0281	6.82	7.01	1.0346	8.36	8.64	1.0411	9.87	10.28	1.0476	11.37	11.91
1.0282	6.85	7.04	1.0347	8.38	8.67	1.0412	9.89	10.30	1.0477	11.39	11.94
1.0283	6.87	7.06	1.0348	8.40	8.69	1.0413	9.92	10.33	1.0478	11.42	11.96
1.0284	6.89	7.09	1.0349	8.43	8.72	1.0414	9.94	10.35	1.0479	11.44	11.99
1.0285	6.92	7.11	1.0350	8.45	8.74	1.0415	9.96	10.38	1.0480	11.46	12.01
1.0286	6.94	7.14	1.0351	8.47	8.77	1.0416	9.99	10.40	1.0481	11.48	12.04
1.0287	6.97	7.16	1.0352	8.50	8.79	1.0417	10.01	10.43	1.0482	11.51	12.06
1.0288	6.99	7.19	1.0353	8.52	8.82	1.0418	10.03	10.45	1.0488	11.53	12.09
1.0289	7.01	7.22	1.0354	8.54	8.84	1.0419	10.06	10.48	1.0484	11.55	12.11
1.0290	7.04	7.24	1.0355	8.57	8.87	1.0420	10.08	10.50	1.0485	11.58	12.14
1.0291	7.06	7.27	1.0356	8.59	8.90	1.0421	10.10	10.53	1.0486	11.60	12.16
1.0292	7.08	7.29	1.0357	8.61	8.92	1.0422	10.13	10.55	1.0487	11.62	12.19
1.0293	7.11	7.32	1.0358	8.64	8.95	1.0423	10.15	10.58	1.0488	11.65	12.21
1.0294	7.13	7.34	1.0359	8.66	8.97	1.0424	10.17	10.60	1.0489	11.67	12.24
1.0295	7.15	7.37	1.0360	8.68	9.00	1.0425	10.20	10.63	1.0490	11.69	12.26
1.0296	7.18	7.39	1.0361	8.71	9.02	1.0426	10.22	10.65	1.0491	11.71	12.29
1.0297	7.20	7.42	1.0362	8.73	9.05	1.0427	10.24	10.68	1.0492	11.74	12.31
1.0298	7.23	7.44	1.0363	8.75	9.07	1.0428	10.26	10.70	1.0493	11.76	12.34
1.0299	7.25	7.47	1.0364	8.78	9.10	1.0429	10.29	10.73	1.0494	11.78	12.36
1.0300	7.27	7.49	1.0365	8.80	9.12	1.0430	10.31	10.75	1.0495	11.81	12.39
1.0301	7.30	7.52	1.0366	8.82	9.15	1.0431	10.33	10.78	1.0496	11.83	12.42
1.0302	7.32	7.54	1.0367	8.85	9.17	1.0432	10.36	10.80	1.0497	11.85	12.44
1.0303	7.34	7.57	1.0368	8.87	9.20	1.0433	10.38	10.83	1.0498	11.87	12.47
1.0304	7.37	7.59	1.0369	8.89	9.22	1.0434	10.40	10.85	1.0499	11.90	12.49
1.0305	7.39	7.62	1.0370	8.92	9.25	1.0435	10.43	10.88	1.0500	11.92	12.52
1.0306	7.41	7.64	1.0371	8.94	9.27	1.0436	10.45	10.90	1.0501	11.94	12.54
1.0307	7.44	7.67	1.0372	8.96	9.30	1.0437	10.47	10.93	1.0502	11.97	12.57
1.0308	7.46	7.69	1.0373	8.99	9.32	1.0438	10.50	10.96	1.0503	11.99	12.59
1.0309	7.49	7.72	1.0374	9.01	9.35	1.0439	10.52	10.98	1.0504	12.01	12.62
1.0310	7.51	7.74	1.0375	9.03	9.37	1.0440	10.54	11.01	1.0505	12.03	12.64
1.0311	7.53	7.77	1.0376	9.06	9.40	1.0441	10.56	11.03	1.0506	12.06	12.67
1.0312	7.56	7.79	1.0377	9.08	9.42	1.0442	10.59	11.06	1.0507	12.08	12.69
1.0313	7.58	7.82	1.0378	9.10	9.45	1.0443	10.61	11.08	1.0508	12.10	12.72
1.0314	7.60	7.84	1.0379	9.13	9.47	1.0444	10.63	11.11	1.0509	12.13	12.74
1.0315	7.63	7.87	1.0380	9.15	9.50	1.0445	10.66	11.13	1.0510	12.15	12.77
1.0316	7.65	7.89	1.0381	9.17	9.52	1.0446	10.68	11.16	1.0511	12.17	12.79
1.0317	7.67	7.92	1.0382	9.20	9.55	1.0447	10.70	11.18	1.0512	12.19	12.82
1.0318	7.70	7.94	1.0383	9.22	9.57	1.0448	10.73	11.21	1.0513	12.22	12.84
1.0319	7.72	7.97	1.0384	9.24	9.60	1.0449	10.75	11.23	1.0514	12.24	12.87
1.0320	7.74	7.99	1.0385	9.27	9.62	1.0450	10.77	11.26	1.0515	12.26	12.89
1.0321	7.77	8.02	1.0386	9.29	9.65	1.0451	10.80	11.28	1.0516	12.28	12.92
1.0322	7.79	8.04	1.0387	9.31	9.67	1.0452	10.82	11.31	1.0517	12.31	12.94
1.0323	7.81	8.07	1.0388	9.34	9.70	1.0453	10.84	11.33	1.0518	12.33	12.97
1.0324	7.84	8.09	1.0389	9.36	9.72	1.0454	10.86	11.36	1.0519	12.35	12.99
1.0325	7.86	8.12	1.0390	9.38	9.75	1.0455	10.88	11.38	1.0520	12.38	13.02

TABLE IV.—*Extract in beer wort*—Continued.

Specific gravity at 15°C.	Extract.		Specific gravity at 15°C.	Extract.		Specific gravity at 15°C.	Extract.		Specific gravity at 15°C.	Extract.	
	Per cent by weight.	Grams per 100 cc.		Per cent by weight.	Grams per 100 cc.		Per cent by weight.	Grams per 100 cc.		Per cent by weight.	Grams per 100 cc.
1.0521	12.40	13.04	1.0586	13.87	14.68	1.0651	15.33	16.33	1.0716	16.77	17.97
1.0522	12.42	13.07	1.0587	13.89	14.71	1.0652	15.35	16.35	1.0717	16.97	18.00
1.0523	12.44	13.10	1.0588	13.92	14.73	1.0653	15.37	16.38	1.0718	16.82	18.02
1.0524	12.47	13.12	1.0589	15.94	14.76	1.0654	15.40	16.40	1.0719	16.84	18.05
1.0525	12.49	13.15	1.0590	13.96	14.79	1.0655	15.42	16.43	1.0720	16.86	18.07
1.0526	12.51	13.17	1.0591	13.98	14.81	1.0656	15.44	16.45	1.0721	16.88	18.10
1.0527	12.54	13.20	1.0592	14.01	14.84	1.0657	15.46	16.48	1.0722	16.90	18.12
1.0528	12.56	13.22	1.0593	14.03	14.86	1.0658	15.48	16.50	1.0723	16.93	18.15
1.0529	12.58	13.25	1.0594	14.05	14.89	1.0659	15.51	16.53	1.0724	16.95	18.17
1.0530	12.60	13.27	1.0595	14.07	14.91	1.0660	15.53	16.55	1.0725	16.97	18.20
1.0531	12.63	13.30	1.0596	14.10	14.94	1.0661	15.55	16.58	1.0726	16.99	18.23
1.0532	12.65	13.32	1.0597	14.12	14.96	1.0662	15.57	16.60	1.0727	17.01	18.25
1.0533	12.67	13.35	1.0598	14.14	14.99	1.0663	15.60	16.63	1.0728	17.04	18.28
1.0534	12.69	13.37	1.0599	14.16	15.01	1.0664	15.62	16.66	1.0729	17.06	18.30
1.0535	12.72	13.40	1.0600	14.19	15.04	1.0665	15.64	16.68	1.0730	17.08	18.33
1.0536	12.74	13.42	1.0601	14.21	15.06	1.0666	15.66	16.71	1.0731	17.10	18.35
1.0537	12.76	13.45	1.0602	14.23	15.09	1.0667	15.69	16.73	1.0732	17.12	18.38
1.0538	12.79	13.47	1.0603	14.25	15.11	1.0668	15.71	16.76	1.0733	17.15	18.40
1.0539	12.81	13.50	1.0604	14.28	15.14	1.0669	15.73	16.78	1.0734	17.17	18.43
1.0540	12.83	13.52	1.0605	14.30	15.16	1.0670	15.75	16.81	1.0735	17.19	18.45
1.0541	12.85	13.55	1.0606	14.32	15.19	1.0671	15.77	16.83	1.0736	17.21	18.48
1.0542	12.88	13.57	1.0607	14.34	15.21	1.0672	15.80	16.86	1.0737	17.23	18.50
1.0543	12.90	13.60	1.0608	14.37	15.24	1.0673	15.82	16.88	1.0738	17.26	18.53
1.0544	12.92	13.62	1.0609	14.39	15.27	1.0674	15.84	16.91	1.0739	17.28	18.55
1.0545	12.94	13.65	1.0610	14.41	15.29	1.0675	15.86	16.93	1.0740	17.30	18.58
1.0546	12.97	13.68	1.0611	14.43	15.32	1.0676	15.89	16.96	1.0741	17.32	18.61
1.0547	12.99	13.70	1.0612	14.46	15.34	1.0677	15.91	16.98	1.0742	17.34	18.63
1.0548	13.01	13.73	1.0613	14.48	15.37	1.0678	15.93	17.01	1.0743	17.37	18.66
1.0549	13.04	13.75	1.0614	14.50	15.39	1.0679	15.95	17.03	1.0744	17.39	18.68
1.0550	13.06	13.78	1.0615	14.52	15.42	1.0680	15.97	17.06	1.0745	17.41	18.71
1.0551	13.08	13.80	1.0616	14.55	15.44	1.0681	16.00	17.09	1.0746	17.43	18.73
1.0552	13.10	13.83	1.0617	14.57	15.47	1.0682	16.02	17.11	1.0747	17.45	18.76
1.0553	13.13	13.85	1.0618	14.59	15.49	1.0683	16.04	17.14	1.0748	17.48	18.78
1.0554	13.15	13.88	1.0619	14.61	15.52	1.0684	16.06	17.16	1.0749	17.50	18.81
1.0555	13.17	13.90	1.0620	14.64	15.54	1.0685	16.09	17.19	1.0750	17.52	18.83
1.0556	13.19	13.93	1.0621	14.66	15.57	1.0686	16.11	17.21	1.0751	17.54	18.86
1.0557	13.22	13.95	1.0622	14.68	15.59	1.0687	16.13	17.24	1.0752	17.56	18.88
1.0558	13.24	13.98	1.0623	14.70	15.62	1.0688	16.15	17.26	1.0753	17.59	18.91
1.0559	13.26	14.00	1.0624	14.73	15.64	1.0689	16.17	17.29	1.0754	17.61	18.93
1.0560	13.28	14.03	1.0625	14.75	15.67	1.0690	16.20	17.31	1.0755	17.63	18.96
1.0561	13.31	14.05	1.0626	14.77	15.69	1.0691	16.22	17.34	1.0756	17.65	18.99
1.0562	13.33	14.08	1.0627	14.79	15.72	1.0692	16.24	17.36	1.0757	17.67	19.01
1.0563	13.35	14.10	1.0628	14.81	15.75	1.0693	16.26	17.39	1.0758	17.69	19.04
1.0564	13.37	14.13	1.0629	14.84	15.77	1.0694	16.28	17.41	1.0759	17.72	19.06
1.0565	13.40	14.15	1.0630	14.86	15.80	1.0695	16.31	17.44	1.0760	17.74	19.09
1.0566	13.42	14.18	1.0631	14.88	15.82	1.0696	16.33	17.47	1.0761	17.76	19.11
1.0567	13.44	14.20	1.0632	14.90	15.85	1.0697	16.35	17.49	1.0762	17.78	19.14
1.0568	13.47	14.23	1.0633	14.93	15.87	1.0698	16.37	17.52	1.0763	17.80	19.16
1.0569	13.49	14.26	1.0634	14.95	15.90	1.0699	16.40	17.54	1.0764	17.83	19.19
1.0570	13.51	14.28	1.0635	14.97	15.92	1.0700	16.42	17.57	1.0765	17.85	19.21
1.0571	13.53	14.31	1.0636	14.99	15.95	1.0701	16.44	17.59	1.0766	17.87	19.24
1.0572	13.56	14.33	1.0637	15.02	15.97	1.0702	16.46	17.62	1.0767	17.89	19.26
1.0573	13.58	14.36	1.0638	15.04	16.00	1.0703	16.48	17.64	1.0768	17.91	19.29
1.0574	13.60	14.38	1.0639	15.06	16.02	1.0704	16.51	17.67	1.0769	17.94	19.32
1.0575	13.62	14.41	1.0640	15.08	16.05	1.0705	16.53	17.69	1.0770	17.96	19.34
1.0576	13.65	14.43	1.0641	15.11	16.07	1.0706	16.55	17.72	1.0771	17.98	19.37
1.0577	13.67	14.46	1.0642	15.13	16.10	1.0707	16.57	17.74	1.0772	18.00	19.39
1.0578	13.69	14.48	1.0643	15.15	16.12	1.0708	16.59	17.77	1.0773	18.02	19.42
1.0579	13.71	14.51	1.0644	15.17	16.15	1.0709	16.62	17.79	1.0774	18.05	19.44
1.0580	13.74	14.53	1.0645	15.19	16.18	1.0710	16.64	17.82	1.0775	18.07	19.47
1.0581	13.76	14.56	1.0646	15.22	16.20	1.0711	16.66	17.85	1.0776	18.09	19.49
1.0582	13.78	14.58	1.0647	15.24	16.23	1.0712	16.68	17.87	1.0777	18.11	19.52
1.0583	13.80	14.61	1.0648	15.26	16.25	1.0713	16.70	17.90	1.0778	18.13	19.54
1.0584	13.83	14.63	1.0649	15.28	16.28	1.0714	16.73	17.92	1.0779	18.15	19.57
1.0585	13.85	14.66	1.0650	15.31	16.30	1.0715	16.75	17.95	1.0780	18.18	19.59



TABLE IV.—*Extract in beer wort*—Continued.

Specific gravity at 15° C.	Extract.		Specific gravity at 15° C.	Extract.		Specific gravity at 15° C.	Extract.		Specific gravity at 15° C.	Extract.	
	Per cent by weight.	Grams per 100 cc.		Per cent by weight.	Grams per 100 cc.		Per cent by weight.	Grams per 100 cc.		Per cent by weight.	Grams per 100 cc.
1.0781	18.20	19.62	1.0836	19.39	21.02	1.0891	20.58	22.41	1.0946	21.76	23.81
1.0782	18.22	19.64	1.0837	19.42	21.04	1.0892	20.60	22.44	1.0947	21.78	23.84
1.0783	18.24	19.67	1.0838	19.44	21.07	1.0893	20.62	22.47	1.0948	21.80	23.87
1.0784	18.26	19.70	1.0839	19.46	21.09	1.0894	20.65	22.49	1.0949	21.82	23.89
1.0785	18.29	19.72	1.0840	19.48	21.12	1.0895	20.67	22.52	1.0950	21.84	23.92
1.0786	18.31	19.75	1.0841	19.50	21.14	1.0896	20.69	22.54	1.0951	21.86	23.94
1.0787	18.33	19.77	1.0842	19.52	21.17	1.0897	20.71	22.57	1.0952	21.88	23.97
1.0788	18.35	19.80	1.0843	19.55	21.19	1.0898	20.73	22.59	1.0953	21.91	23.99
1.0789	18.37	19.82	1.0844	19.57	21.22	1.0899	20.75	22.62	1.0954	21.93	24.02
1.0790	18.39	19.85	1.0845	19.59	21.24	1.0900	20.77	22.64	1.0955	21.95	24.04
1.0791	18.42	19.87	1.0846	19.61	21.27	1.0901	20.79	22.67	1.0956	21.97	24.07
1.0792	18.44	19.90	1.0847	19.63	21.30	1.0902	20.82	22.69	1.0957	21.99	24.09
1.0793	18.46	19.92	1.0848	19.65	21.32	1.0903	20.84	22.72	1.0958	22.01	24.12
1.0794	18.48	19.95	1.0849	19.68	21.35	1.0904	20.86	22.75	1.0959	22.03	24.15
1.0795	18.50	19.97	1.0850	69.70	21.37	1.0905	20.88	22.77	1.0960	22.05	24.17
1.0796	18.53	20.00	1.0851	19.72	21.40	1.0906	20.90	22.80	1.0961	22.08	24.20
1.0797	18.55	20.03	1.0852	19.74	21.42	1.0907	20.92	22.82	1.0962	22.10	24.22
1.0798	18.57	20.05	1.0853	19.76	21.45	1.0908	20.94	22.85	1.0963	22.12	24.25
1.0799	18.59	20.08	1.0854	19.78	21.47	1.0909	20.97	22.87	1.0964	22.14	24.27
1.0800	18.61	20.10	1.0855	19.81	21.50	1.0910	20.99	22.90	1.0965	22.16	24.30
1.0801	18.63	20.13	1.0856	19.83	21.52	1.0911	21.01	22.92	1.0966	22.18	24.32
1.0802	18.66	20.15	1.0857	19.85	21.55	1.0912	21.03	22.95	1.0967	22.20	24.35
1.0803	18.68	20.18	1.0858	19.87	21.58	1.0913	21.05	22.97	1.0968	22.22	24.38
1.0804	18.70	20.20	1.0859	19.89	21.60	1.0914	21.07	23.00	1.0969	22.25	24.40
1.0805	18.72	20.23	1.0860	19.91	21.63	1.0915	21.09	23.02	1.0970	22.27	24.43
1.0806	18.74	20.25	1.0861	19.93	21.65	1.0916	21.12	23.05	1.0971	22.29	24.45
1.0807	18.77	20.28	1.0862	19.96	21.68	1.0917	21.14	23.08	1.0972	22.31	24.48
1.0808	18.79	20.30	1.0863	19.98	21.70	1.0918	21.16	23.10	1.0973	22.33	24.50
1.0809	18.81	20.33	1.0864	20.00	21.73	1.0919	21.18	23.13	1.0974	22.35	24.53
1.0810	18.83	20.36	1.0865	20.02	21.75	1.0920	21.20	23.15	1.0975	22.37	24.55
1.0811	18.85	20.38	1.0866	20.04	21.78	1.0921	21.22	23.18	1.0976	22.39	24.58
1.0812	18.87	20.41	1.0867	20.06	21.80	1.0922	21.24	23.20	1.0977	22.41	24.60
1.0813	18.90	20.43	1.0868	20.09	21.83	1.0923	21.27	23.23	1.0978	22.44	24.63
1.0814	18.92	20.46	1.0869	20.11	21.85	1.0924	21.29	23.25	1.0979	22.46	24.66
1.0815	18.94	20.48	1.0870	20.13	21.88	1.0925	21.31	33.28	1.0980	22.48	24.68
1.0816	18.96	20.51	1.0871	20.15	21.91	1.0926	21.33	23.31	1.0981	22.50	24.71
1.0817	18.98	20.53	1.0872	20.17	21.93	1.0927	21.35	23.33	1.0982	22.52	24.73
1.0818	19.00	20.56	1.0873	20.19	21.96	1.0928	21.37	23.36	1.0983	22.54	24.76
1.0819	19.03	20.58	1.0874	25.21	21.98	1.0929	21.39	23.38	1.0984	22.56	25.78
1.0820	19.05	20.61	1.0875	20.24	22.01	1.0930	21.42	23.41	1.0985	22.58	24.81
1.0821	19.07	20.63	1.0876	20.26	22.03	1.0931	21.44	23.43	1.0986	22.61	24.83
1.0822	19.09	20.66	1.0877	20.28	22.06	1.0932	21.46	23.46	1.0987	22.63	24.86
1.0823	19.11	20.69	1.0878	20.30	22.08	1.0933	21.48	23.48	1.0988	22.65	24.89
1.0824	19.13	20.71	1.0879	20.32	22.11	1.0934	21.50	23.51	1.0989	22.67	24.91
1.0825	19.16	20.74	1.0880	20.34	22.13	1.0935	11.52	23.53	1.0990	22.69	24.94
1.0826	19.18	20.76	1.0881	20.37	22.16	1.0936	21.54	23.56	1.0991	22.71	24.96
1.0827	19.20	20.79	1.0882	10.39	22.19	1.0937	21.56	23.59	1.0992	22.73	24.99
1.0828	19.22	20.81	1.0883	20.41	22.21	1.0938	21.59	23.61			
1.0829	19.24	20.84	1.0884	20.43	22.24	1.0939	21.61	23.64			
1.0830	19.26	20.86	1.0885	20.45	22.26	1.0940	21.63	23.66			
1.0831	19.29	20.89	1.0886	20.47	22.29	1.0941	21.65	23.69			
1.0832	19.31	20.91	1.0887	20.49	22.31	1.0942	21.67	23.71			
1.0833	19.33	20.94	1.0888	20.52	22.34	1.0943	21.69	23.74			
1.0834	19.35	20.96	1.0889	20.54	22.36	1.0944	21.71	23.76			
1.0835	19.37	20.99	1.0890	20.56	22.39	1.0945	21.73	23.79			

TABLE V.—*Extract in wine.*

[According to Windisch.]

Specific gravity.	Ex-tract.	Specific gravity.	Ex-tract.	Specific gravity.	Ex-tract.	Specific gravity.	Ex-tract.	Specific gravity.	Ex-tract.	Specific gravity.	Ex-tract.
1.0000	0.00	1.0065	1.68	1.0130	3.36	1.0195	5.04	1.0260	6.72	1.0325	8.40
1.0001	0.03	1.0066	1.70	1.0131	3.38	1.0196	5.06	1.0261	6.75	1.0326	8.43
1.0002	0.05	1.0067	1.73	1.0132	3.41	1.0197	5.09	1.0262	6.77	1.0327	8.46
1.0003	0.08	1.0068	1.76	1.0133	3.43	1.0198	5.11	1.0263	6.80	1.0328	8.48
1.0004	0.10	1.0069	1.78	1.0134	3.46	1.0199	5.14	1.0264	6.82	1.0329	8.51
1.0005	0.13	1.0070	1.81	1.0135	3.49	1.0200	5.17	1.0265	6.85	1.0330	8.53
1.0006	0.15	1.0071	1.83	1.0136	3.51	1.0201	5.19	1.0266	6.88	1.0331	8.56
1.0007	0.18	1.0072	1.86	1.0137	3.54	1.0202	5.22	1.0267	6.90	1.0332	8.59
1.0008	0.20	1.0073	1.88	1.0138	3.56	1.0203	5.25	1.0268	6.93	1.0333	8.61
1.0009	0.23	1.0074	1.91	1.0139	3.59	1.0204	5.27	1.0269	6.95	1.0334	8.64
1.0010	0.26	1.0075	1.94	1.0140	3.62	1.0205	5.30	1.0270	6.98	1.0335	8.66
1.0011	0.28	1.0076	1.96	1.0141	3.64	1.0206	5.32	1.0271	7.01	1.0336	8.69
1.0012	0.31	1.0077	1.99	1.0142	3.67	1.0207	5.35	1.0272	7.03	1.0337	8.72
1.0013	0.34	1.0078	2.01	1.0143	3.69	1.0208	5.38	1.0273	7.06	1.0338	8.74
1.0014	0.36	1.0079	2.04	1.0144	3.72	1.0209	5.40	1.0274	7.08	1.0339	8.77
1.0015	0.39	1.0080	2.07	1.0145	3.75	1.0210	5.43	1.0275	7.11	1.0340	8.79
1.0016	0.41	1.0081	2.09	1.0146	3.77	1.0211	5.45	1.0276	7.13	1.0341	8.82
1.0017	0.44	1.0082	2.12	1.0147	3.80	1.0212	5.48	1.0277	7.16	1.0342	8.85
1.0018	0.46	1.0083	2.14	1.0148	3.82	1.0213	5.51	1.0278	7.19	1.0343	8.87
1.0019	0.49	1.0084	2.17	1.0149	3.85	1.0214	5.53	1.0279	7.21	1.0344	8.90
1.0020	0.52	1.0085	2.19	1.0150	3.87	1.0215	5.56	1.0280	7.24	1.0345	8.92
1.0021	0.54	1.0086	2.22	1.0151	3.90	1.0216	5.58	1.0281	7.26	1.0346	8.95
1.0022	0.57	1.0087	2.25	1.0152	3.93	1.0217	5.61	1.0282	7.29	1.0347	8.97
1.0023	0.59	1.0088	2.27	1.0153	3.95	1.0218	5.64	1.0283	7.32	1.0348	9.00
1.0024	0.62	1.0089	2.30	1.0154	3.98	1.0219	5.66	1.0284	7.34	1.0349	9.03
1.0025	0.64	1.0090	2.32	1.0155	4.00	1.0220	5.69	1.0285	7.37	1.0350	9.05
1.0026	0.67	1.0091	2.35	1.0156	4.03	1.0221	5.71	1.0286	7.39	1.0351	9.08
1.0027	0.69	1.0092	2.38	1.0157	4.06	1.0222	5.74	1.0287	7.42	1.0352	9.10
1.0028	0.72	1.0093	2.40	1.0158	4.08	1.0223	5.77	1.0288	7.45	1.0353	9.13
1.0029	0.75	1.0094	2.43	1.0159	4.11	1.0224	5.79	1.0289	7.47	1.0354	9.16
1.0030	0.77	1.0095	2.45	1.0160	4.13	1.0225	5.82	1.0290	7.50	1.0355	9.18
1.0031	0.80	1.0096	2.48	1.0161	4.16	1.0226	5.84	1.0291	7.52	1.0356	9.21
1.0032	0.82	1.0097	2.50	1.0162	4.19	1.0227	5.87	1.0292	7.55	1.0357	9.23
1.0033	0.85	1.0098	2.53	1.0163	4.21	1.0228	5.89	1.0293	7.58	1.0358	9.26
1.0034	0.87	1.0099	2.56	1.0164	4.24	1.0229	5.92	1.0294	7.60	1.0359	9.29
1.0035	0.90	1.0100	2.58	1.0165	4.26	1.0230	5.94	1.0295	7.63	1.0360	9.31
1.0036	0.93	1.0101	2.61	1.0166	4.29	1.0231	5.97	1.0296	7.65	1.0361	9.34
1.0037	0.95	1.0102	2.63	1.0167	4.31	1.0232	6.00	1.0297	7.68	1.0362	9.36
1.0038	0.98	1.0103	2.66	1.0168	4.34	1.0233	6.02	1.0298	7.70	1.0363	9.39
1.0039	1.00	1.0104	2.69	1.0169	4.37	1.0234	6.05	1.0299	7.73	1.0364	9.42
1.0040	1.03	1.0105	2.71	1.0170	4.39	1.0235	6.07	1.0300	7.76	1.0365	9.44
1.0041	1.05	1.0106	2.74	1.0171	4.42	1.0236	6.10	1.0301	7.78	1.0366	9.47
1.0042	1.08	1.0107	2.76	1.0172	4.44	1.0237	6.12	1.0302	7.81	1.0367	9.49
1.0043	1.11	1.0108	2.79	1.0173	4.47	1.0238	6.15	1.0303	7.83	1.0368	9.52
1.0044	1.13	1.0109	2.82	1.0174	4.50	1.0239	6.18	1.0304	7.86	1.0369	9.55
1.0045	1.16	1.0110	2.84	1.0175	4.52	1.0240	6.20	1.0305	7.89	1.0370	9.57
1.0046	1.18	1.0111	2.87	1.0176	4.55	1.0241	6.23	1.0306	7.91	1.0371	9.60
1.0047	1.21	1.0112	2.89	1.0177	4.57	1.0242	6.25	1.0307	7.94	1.0372	9.62
1.0048	1.24	1.0113	2.92	1.0178	4.60	1.0243	6.28	1.0308	7.97	1.0373	9.65
1.0049	1.26	1.0114	2.94	1.0179	4.63	1.0244	6.31	1.0309	7.99	1.0374	9.68
1.0050	1.29	1.0115	2.97	1.0180	4.65	1.0245	6.33	1.0310	8.02	1.0375	9.70
1.0051	1.32	1.0116	3.00	1.0181	4.68	1.0246	6.36	1.0311	8.04	1.0376	9.73
1.0052	1.34	1.0117	3.02	1.0182	4.70	1.0247	6.38	1.0312	8.07	1.0377	9.75
1.0053	1.37	1.0118	3.05	1.0183	4.73	1.0248	6.41	1.0313	8.09	1.0378	9.78
1.0054	1.39	1.0119	3.07	1.0184	4.75	1.0249	6.44	1.0314	8.12	1.0379	9.80
1.0055	1.42	1.0120	3.10	1.0185	4.78	1.0250	6.46	1.0315	8.14	1.0380	9.83
1.0056	1.45	1.0121	3.12	1.0186	4.81	1.0251	6.49	1.0316	8.17	1.0381	9.86
1.0057	1.47	1.0122	3.15	1.0187	4.83	1.0252	6.51	1.0317	8.20	1.0382	9.88
1.0058	1.50	1.0123	3.18	1.0188	4.86	1.0253	6.54	1.0318	8.22	1.0383	9.91
1.0059	1.52	1.0124	3.20	1.0189	4.88	1.0254	6.56	1.0319	8.25	1.0384	9.93
1.0060	1.55	1.0125	3.23	1.0190	4.91	1.0255	6.59	1.0320	8.27	1.0385	9.96
1.0061	1.57	1.0126	3.26	1.0191	4.94	1.0256	6.62	1.0321	8.30	1.0386	9.99
1.0062	1.60	1.0127	3.28	1.0192	4.96	1.0257	6.64	1.0322	8.33	1.0387	10.01
1.0063	1.63	1.0128	3.31	1.0193	4.99	1.0258	6.67	1.0323	8.35	1.0388	10.04
1.0064	1.65	1.0129	3.33	1.0194	5.01	1.0259	6.70	1.0324	8.38	1.0389	10.06

TABLE V.—*Extract in wine*—Continued.

Specific gravity.	Ex-tract.	Specific gravity.	Ex-tract.	Specific gravity.	Ex-tract.	Specific gravity.	Ex-tract.	Specific gravity.	Ex-tract.	Specific gravity.	Ex-tract.
1.0390	10.09	1.0455	11.78	1.0520	13.47	1.0585	15.16	1.0650	16.86	1.0715	18.56
1.0391	10.11	1.0456	11.81	1.0521	13.49	1.0586	15.19	1.0651	16.88	1.0716	18.58
1.0392	10.14	1.0457	11.83	1.0522	13.52	1.0587	15.22	1.0652	16.91	1.0717	18.61
1.0393	10.17	1.0458	11.86	1.0523	13.55	1.0588	15.24	1.0653	16.94	1.0718	18.63
1.0394	10.19	1.0459	11.88	1.0524	13.57	1.0589	15.27	1.0654	16.96	1.0719	18.66
1.0395	10.22	1.0460	11.91	1.0525	13.60	1.0590	15.29	1.0655	16.99	1.0720	18.69
1.0396	10.25	1.0461	11.94	1.0526	13.62	1.0591	15.32	1.0656	17.01	1.0721	18.71
1.0397	10.27	1.0462	11.96	1.0527	13.65	1.0592	15.35	1.0657	17.04	1.0722	18.74
1.0398	10.30	1.0463	11.99	1.0528	13.68	1.0593	15.37	1.0658	17.07	1.0723	18.76
1.0399	10.32	1.0464	12.01	1.0529	13.70	1.0594	15.40	1.0659	17.09	1.0724	18.79
1.0400	10.35	1.0465	12.04	1.0530	13.73	1.0595	15.42	1.0660	17.12	1.0725	18.82
1.0401	10.37	1.0466	12.06	1.0531	13.75	1.0596	15.45	1.0661	17.14	1.0726	18.84
1.0402	10.40	1.0467	12.09	1.0532	13.78	1.0597	15.48	1.0662	17.17	1.0727	18.87
1.0403	10.43	1.0468	12.12	1.0533	13.81	1.0598	15.50	1.0663	17.20	1.0728	18.90
1.0404	10.45	1.0469	12.14	1.0534	13.83	1.0599	15.53	1.0664	17.22	1.0729	18.92
1.0405	10.48	1.0470	12.17	1.0535	13.86	1.0600	15.55	1.0665	17.25	1.0730	18.95
1.0406	10.51	1.0471	12.19	1.0536	13.89	1.0601	15.58	1.0666	17.27	1.0731	18.97
1.0407	10.53	1.0472	12.22	1.0537	13.91	1.0602	15.61	1.0667	17.30	1.0732	19.00
1.0408	10.56	1.0473	12.25	1.0538	13.94	1.0603	15.63	1.0668	17.33	1.0733	19.03
1.0409	10.58	1.0474	12.27	1.0539	13.96	1.0604	15.66	1.0669	17.35	1.0734	19.05
1.0410	10.61	1.0475	12.30	1.0540	13.99	1.0605	15.68	1.0670	17.38	1.0735	19.08
1.0411	10.63	1.0476	12.32	1.0541	14.01	1.0606	15.71	1.0671	17.41	1.0736	19.10
1.0412	10.66	1.0477	12.35	1.0542	14.04	1.0607	15.74	1.0672	17.43	1.0737	19.13
1.0413	10.69	1.0478	12.38	1.0543	15.07	1.0608	15.76	1.0673	17.46	1.0738	19.16
1.0414	10.71	1.0479	12.40	1.0544	14.09	1.0609	15.79	1.0674	17.48	1.0739	19.18
1.0415	10.74	1.0480	12.43	1.0545	14.12	1.0610	15.81	1.0675	17.51	1.0740	19.21
1.0416	10.76	1.0481	12.45	1.0546	14.14	1.0611	15.84	1.0676	17.54	1.0741	19.23
1.0417	10.79	1.0482	12.48	1.0547	14.17	1.0612	15.87	1.0677	17.56	1.0742	19.26
1.0418	10.82	1.0483	12.51	1.0548	14.20	1.0613	15.89	1.0678	17.59	1.0743	19.29
1.0419	10.84	1.0484	12.53	1.0549	14.22	1.0614	15.92	1.0679	17.62	1.0744	19.31
1.0420	10.87	1.0485	12.56	1.0550	14.25	1.0615	15.94	1.0680	17.64	1.0745	19.34
1.0421	10.90	1.0486	12.58	1.0551	14.28	1.0616	15.97	1.0681	17.67	1.0746	19.37
1.0422	10.92	1.0487	12.61	1.0552	14.30	1.0617	16.00	1.0682	17.69	1.0747	19.39
1.0423	10.95	1.0488	12.64	1.0553	14.33	1.0618	16.02	1.0683	17.72	1.0748	19.42
1.0424	10.97	1.0489	12.66	1.0554	14.35	1.0619	16.05	1.0684	17.75	1.0749	19.44
1.0425	11.00	1.0490	12.69	1.0555	14.38	1.0620	16.07	1.0685	17.77	1.0750	19.47
1.0426	11.03	1.0491	12.71	1.0556	14.41	1.0621	16.10	1.0686	17.80	1.0751	19.50
1.0427	11.05	1.0492	12.74	1.0557	14.43	1.0622	16.13	1.0687	17.83	1.0752	19.52
1.0428	11.08	1.0493	12.77	1.0558	14.46	1.0623	16.15	1.0688	17.85	1.0753	19.55
1.0429	11.10	1.0494	12.79	1.0559	14.48	1.0624	16.18	1.0689	17.88	1.0754	19.58
1.0430	11.13	1.0495	12.82	1.0560	14.51	1.0625	16.21	1.0690	17.90	1.0755	19.60
1.0431	11.15	1.0496	12.84	1.0561	14.54	1.0626	16.23	1.0691	17.93	1.0756	19.63
1.0432	11.18	1.0497	12.87	1.0562	14.56	1.0627	16.26	1.0692	17.95	1.0757	19.65
1.0433	11.21	1.0498	12.90	1.0563	14.59	1.0628	16.28	1.0693	17.98	1.0758	19.68
1.0434	11.23	1.0499	12.92	1.0564	14.61	1.0629	16.31	1.0694	18.01	1.0759	19.71
1.0435	11.26	1.0500	12.95	1.0565	14.64	1.0630	16.33	1.0695	18.03	1.0760	19.73
1.0436	11.28	1.0501	12.97	1.0566	14.67	1.0631	16.36	1.0696	18.06	1.0761	19.76
1.0437	11.31	1.0502	13.00	1.0567	14.69	1.0632	16.39	1.0697	18.08	1.0762	19.79
1.0438	11.34	1.0503	13.03	1.0568	14.72	1.0633	16.41	1.0698	18.11	1.0763	19.81
1.0439	11.36	1.0504	13.05	1.0569	14.74	1.0634	16.44	1.0699	18.14	1.0764	19.84
1.0440	11.39	1.0505	13.08	1.0570	14.77	1.0635	16.47	1.0700	18.16	1.0765	19.86
1.0441	11.42	1.0506	13.10	1.0571	14.80	1.0636	16.49	1.0701	18.19	1.0766	19.89
1.0442	11.44	1.0507	13.13	1.0572	14.82	1.0637	16.52	1.0702	18.22	1.0767	19.92
1.0443	11.47	1.0508	13.16	1.0573	14.85	1.0638	16.54	1.0703	18.24	1.0768	19.94
1.0444	11.49	1.0509	13.18	1.0574	14.87	1.0639	16.57	1.0704	18.27	1.0769	19.97
1.0445	11.52	1.0510	13.21	1.0575	14.90	1.0640	16.60	1.0705	18.30	1.0770	20.00
1.0446	11.55	1.0511	13.23	1.0576	14.93	1.0641	16.62	1.0706	18.32	1.0771	20.02
1.0447	11.57	1.0512	13.26	1.0577	14.95	1.0642	16.65	1.0707	18.35	1.0772	20.05
1.0448	11.60	1.0513	13.29	1.0578	14.98	1.0643	16.68	1.0708	18.37	1.0773	20.07
1.0449	11.62	1.0514	13.31	1.0579	15.00	1.0644	16.70	1.0709	18.40	1.0774	20.10
1.0450	11.65	1.0515	13.34	1.0580	15.03	1.0645	16.73	1.0710	18.43	1.0775	20.12
1.0451	11.68	1.0516	13.36	1.0581	15.06	1.0646	16.75	1.0711	18.45	1.0776	20.15
1.0452	11.70	1.0517	13.39	1.0582	15.08	1.0647	16.78	1.0712	18.48	1.0777	20.18
1.0453	11.73	1.0518	13.42	1.0583	15.11	1.0648	16.80	1.0713	18.50	1.0778	20.20
1.0454	11.75	1.0519	13.44	1.0584	15.14	1.0649	16.83	1.0714	18.53	1.0779	20.23

TABLE V.—*Extract in wine*—Continued.

Specific gravity.	Ex-tract.	Specific gravity.	Ex-tract.	Specific gravity.	Ex-tract.	Specific gravity.	Ex-tract.	Specific gravity.	Ex-tract.	Specific gravity.	Ex-tract.
1.0780	20.26	1.0845	21.96	1.0910	23.67	1.0975	25.38	1.1040	27.09	1.1105	28.81
1.0781	20.28	1.0846	21.99	1.0911	23.70	1.0976	25.41	1.1041	27.12	1.1106	28.83
1.0782	20.31	1.0847	22.02	1.0912	23.72	1.0977	25.43	1.1042	27.15	1.1107	28.86
1.0783	20.34	1.0848	22.04	1.0913	23.75	1.0978	25.46	1.1043	27.17	1.1108	28.88
1.0784	20.36	1.0849	22.07	1.0914	23.77	1.0979	25.49	1.1044	27.20	1.1109	28.91
1.0785	20.39	1.0850	22.09	1.0915	23.80	1.0980	25.51	1.1045	27.22	1.1110	28.94
1.0786	20.41	1.0851	22.12	1.0916	23.83	1.0981	25.54	1.1046	27.25	1.1111	28.96
1.0787	20.44	1.0852	22.15	1.0917	23.85	1.0982	25.56	1.1047	27.27	1.1112	28.99
1.0788	20.47	1.0853	22.17	1.0918	23.88	1.0983	25.59	1.1048	27.30	1.1113	29.02
1.0789	20.49	1.0854	22.20	1.0919	23.91	1.0984	25.62	1.1049	27.33	1.1114	29.04
1.0790	20.52	1.0855	22.22	1.0920	23.93	1.0985	25.64	1.1050	27.35	1.1115	29.07
1.0791	20.55	1.0856	22.25	1.0921	23.96	1.0986	25.67	1.1051	27.38	1.1116	29.09
1.0792	20.57	1.0857	22.28	1.0922	23.99	1.0987	25.70	1.1052	27.41	1.1117	29.12
1.0793	20.60	1.0858	22.30	1.0923	24.01	1.0988	25.72	1.1053	27.43	1.1118	29.15
1.0794	20.62	1.0859	22.33	1.0924	24.04	1.0989	25.75	1.1054	27.46	1.1119	29.17
1.0795	20.65	1.0860	22.36	1.0925	24.07	1.0990	25.78	1.1055	27.49	1.1120	29.20
1.0796	20.68	1.0861	22.38	1.0926	24.09	1.0991	25.80	1.1056	27.51	1.1121	29.23
1.0797	20.70	1.0862	22.41	1.0927	24.12	1.0992	25.83	1.1057	27.54	1.1122	29.25
1.0798	20.73	1.0863	22.43	1.0928	24.14	1.0993	25.85	1.1058	27.57	1.1123	29.28
1.0799	20.75	1.0864	22.46	1.0929	24.17	1.0994	25.88	1.1059	27.59	1.1124	29.31
1.0800	20.78	1.0865	22.49	1.0930	24.20	1.0995	25.91	1.1060	27.62	1.1125	29.33
1.0801	20.81	1.0866	22.51	1.0931	24.22	1.0996	25.93	1.1061	27.65	1.1126	29.36
1.0802	20.83	1.0867	22.54	1.0932	24.25	1.0997	25.96	1.1062	27.67	1.1127	29.39
1.0803	20.86	1.0868	22.57	1.0933	24.27	1.0998	25.99	1.1063	27.70	1.1128	29.41
1.0804	20.89	1.0869	22.59	1.0934	24.30	1.0999	26.01	1.1064	27.72	1.1129	29.44
1.0805	20.91	1.0870	22.62	1.0935	24.33	1.1000	26.04	1.1065	27.75	1.1130	29.47
1.0806	20.94	1.0871	22.65	1.0936	24.35	1.1001	26.06	1.1066	27.78	1.1131	29.49
1.0807	20.96	1.0872	22.67	1.0937	24.38	1.1002	26.09	1.1067	27.80	1.1132	29.52
1.0808	20.99	1.0873	22.70	1.0938	24.41	1.1003	26.12	1.1068	27.83	1.1133	29.54
1.0809	21.02	1.0874	22.72	1.0939	24.43	1.1004	26.14	1.1069	27.86	1.1134	29.57
1.0810	21.04	1.0875	22.75	1.0940	24.46	1.1005	26.17	1.1070	27.88	1.1135	29.60
1.0811	21.07	1.0876	22.78	1.0941	24.49	1.1006	26.20	1.1071	27.96	1.1136	29.62
1.0812	21.10	1.0877	22.80	1.0942	24.51	1.1007	26.22	1.1072	27.93	1.1137	29.65
1.0813	21.12	1.0878	22.83	1.0943	24.54	1.1008	26.25	1.1073	27.96	1.1138	29.68
1.0814	21.15	1.0879	22.86	1.0944	24.57	1.1009	26.27	1.1074	27.99	1.1139	29.70
1.0815	21.17	1.0880	22.88	1.0945	24.59	1.1010	26.30	1.1075	28.01	1.1140	29.73
1.0816	21.20	1.0881	22.91	1.0946	24.62	1.1011	26.33	1.1076	28.04	1.1141	29.76
1.0817	21.23	1.0882	22.93	1.0947	24.64	1.1012	26.35	1.1077	28.07	1.1142	29.78
1.0818	21.25	1.0883	22.96	1.0948	24.67	1.1013	26.38	1.1078	28.09	1.1143	29.81
1.0819	21.28	1.0884	22.99	1.0949	24.70	1.1014	26.41	1.1079	28.12	1.1144	29.83
1.0820	21.31	1.0885	23.01	1.0950	24.72	1.1015	26.43	1.1080	28.15	1.1145	29.86
1.0821	21.33	1.0886	23.04	1.0951	24.75	1.1016	26.46	1.1081	28.17	1.1146	29.89
1.0822	21.36	1.0887	23.07	1.0952	24.78	1.1017	26.49	1.1082	28.20	1.1147	29.91
1.0823	21.38	1.0888	23.09	1.0953	24.80	1.1018	26.51	1.1083	28.22	1.1148	29.94
1.0824	21.41	1.0889	23.12	1.0954	24.83	1.1019	26.54	1.1084	28.25	1.1149	29.96
1.0825	21.44	1.0890	23.14	1.0955	24.85	1.1020	26.56	1.1085	28.28	1.1150	29.99
1.0826	21.46	1.0891	23.17	1.0956	24.88	1.1021	26.59	1.1056	28.20	1.1151	30.02
1.0827	21.49	1.0892	23.20	1.0957	24.91	1.1022	26.62	1.1087	28.33	1.1152	30.04
1.0828	21.52	1.0893	23.22	1.0958	24.93	1.1023	26.64	1.1088	28.36	1.1153	30.07
1.0829	21.54	1.0894	23.25	1.0959	24.96	1.1024	26.67	1.1089	28.38	1.1154	30.10
1.0830	21.57	1.0895	23.28	1.0960	24.99	1.1025	26.70	1.1090	28.41	1.1155	30.13
1.0831	21.59	1.0896	23.30	1.0961	25.01	1.1026	26.72	1.1091	28.43	1.1156	30.15
1.0832	21.62	1.0897	23.33	1.0962	25.04	1.1027	26.75	1.1092	28.46	1.1157	30.18
1.0833	21.65	1.0898	23.35	1.0963	25.07	1.1028	26.78	1.1093	28.49	1.1158	30.21
1.0834	21.67	1.0899	23.38	1.0964	25.09	1.1029	26.80	1.1094	28.51	1.1159	30.23
1.0835	21.70	1.0900	23.41	1.0965	25.12	1.1030	26.83	1.1095	28.54		
1.0836	21.73	1.0901	23.43	1.0966	25.14	1.1031	26.85	1.1096	28.57		
1.0837	21.75	1.0902	23.46	1.0967	25.17	1.1032	26.88	1.1097	28.59		
1.0838	21.78	1.0903	23.49	1.0968	25.20	1.1033	26.91	1.1098	28.62		
1.0839	21.80	1.0904	23.51	1.0969	25.22	1.1034	26.93	1.1099	28.65		
1.0840	21.83	1.0905	23.54	1.0970	25.25	1.1035	26.96	1.1100	28.67		
1.0841	21.86	1.0906	23.57	1.0971	25.28	1.1036	26.99	1.1101	28.70		
1.0842	21.88	1.0907	23.59	1.0972	25.30	1.1037	27.01	1.1102	28.73		
1.0843	21.91	1.0908	23.62	1.0973	25.33	1.1038	27.04	1.1103	28.75		
1.0844	21.94	1.0909	23.65	1.0974	25.36	1.1039	27.07	1.1104	28.78		

TABLE VI.—*Relation of Brix, specific gravity, and Baumé.*

Per cent of sugar.	Specific gravity.	Degree Baumé.	Per cent of sugar.	Specific gravity.	Degree Baumé.	Per cent of sugar.	Specific gravity.	Degree Baumé.	Per cent of sugar.	Specific gravity.	Degree Baumé.
0.1	1.0003	0.06	6.6	1.0261	3.7	13.1	1.0531	7.3	19.6	1.0815	10.85
0.2	1.0007	0.11	6.7	1.0265	3.7	13.2	1.0536	7.3	19.7	1.0819	10.9
0.3	1.0011	0.17	6.8	1.0269	3.8	13.3	1.0540	7.4	19.8	1.0824	11.0
0.4	1.0015	0.22	6.9	1.0273	3.8	13.4	1.0544	7.4	19.9	1.0828	11.0
0.5	1.0019	0.28	7.0	1.0277	3.9	13.5	1.0548	7.5	20.0	1.0832	11.1
0.6	1.0023	0.33	7.1	1.0281	3.9	13.6	1.0553	7.5	20.1	1.0837	11.1
0.7	1.0027	0.39	7.2	1.0286	4.0	13.7	1.0557	7.6	20.2	1.0841	11.2
0.8	1.0031	0.44	7.3	1.0290	4.1	13.8	1.0561	7.65	20.3	1.0846	11.2
0.9	1.0034	0.5	7.4	1.0294	4.1	13.9	1.0566	7.7	20.4	1.0850	11.3
1.0	1.0038	0.55	7.5	1.0298	4.2	14.0	1.0570	7.8	20.5	1.0855	11.3
1.1	1.0042	0.6	7.6	1.0302	4.2	14.1	1.0574	7.8	20.6	1.0859	11.4
1.2	1.0046	0.7	7.7	1.0306	4.3	14.2	1.0578	7.9	20.7	1.0864	11.45
1.3	1.0050	0.7	7.8	1.0310	4.3	14.3	1.0583	7.9	20.8	1.0868	11.5
1.4	1.0054	0.8	7.9	1.0314	4.4	14.4	1.0587	8.0	20.9	1.0873	11.6
1.5	1.0058	0.8	8.0	1.0318	4.4	14.5	1.0591	8.0	21.0	1.0877	11.6
1.6	1.0062	0.9	8.1	1.0322	4.5	14.6	1.0596	8.1	21.1	1.0882	11.7
1.7	1.0066	0.9	8.2	1.0327	4.55	14.7	1.0600	8.15	21.2	1.0886	11.7
1.8	1.0070	1.0	8.3	1.0331	4.6	14.8	1.0604	8.2	21.3	1.0891	11.8
1.9	1.0074	1.05	8.4	1.0335	4.7	14.9	1.0609	8.3	21.4	1.0895	11.8
2.0	1.0077	1.1	8.5	1.0339	4.7	15.0	1.0613	8.3	21.5	1.0900	11.9
2.1	1.0081	1.2	8.6	1.0343	4.8	15.1	1.0617	8.4	21.6	1.0904	11.95
2.2	1.0085	1.2	8.7	1.0347	4.8	15.2	1.0621	8.4	21.7	1.0909	12.0
2.3	1.0089	1.3	8.8	1.0351	4.9	15.3	1.0626	8.5	21.8	1.0914	12.05
2.4	1.0093	1.3	8.9	1.0355	4.9	15.4	1.0630	8.5	21.9	1.0918	12.1
2.5	1.0097	1.4	9.0	1.0359	5.0	15.5	1.0634	8.6	22.0	1.0923	12.2
2.6	1.0101	1.4	9.1	1.0364	5.05	15.6	1.0639	8.65	22.1	1.0927	12.2
2.7	1.0105	1.5	9.2	1.0368	5.1	15.7	1.0643	8.7	22.2	1.0932	12.3
2.8	1.0109	1.55	9.3	1.0372	5.2	15.8	1.0647	8.8	22.3	1.0936	12.3
2.9	1.0113	1.6	9.4	1.0376	5.2	15.9	1.0652	8.8	22.4	1.0941	12.4
3.0	1.0117	1.7	9.5	1.0380	5.3	16.0	1.0656	8.9	22.5	1.0945	12.4
3.1	1.0121	1.7	9.6	1.0384	5.3	16.1	1.0660	8.9	22.6	1.0950	12.5
3.2	1.0125	1.8	9.7	1.0388	5.4	16.2	1.0665	9.0	22.7	1.0954	12.55
3.3	1.0129	1.8	9.8	1.0393	5.4	16.3	1.0669	9.0	22.8	1.0959	12.6
3.4	1.0133	1.9	9.9	1.0397	5.5	16.4	1.0674	9.1	22.9	1.0964	12.7
3.5	1.0137	1.9	10.0	1.0401	5.55	16.5	1.0678	9.1	23.0	1.0968	12.7
3.6	1.0141	2.0	10.1	1.0405	5.6	16.6	1.0682	9.2	23.1	1.0973	12.8
3.7	1.0145	2.0	10.2	1.0409	5.7	16.7	1.0687	9.25	23.2	1.0977	12.8
3.8	1.0149	2.1	10.3	1.0413	5.7	16.8	1.0691	9.3	23.3	1.0982	12.9
3.9	1.0153	2.2	10.4	1.0418	5.8	16.9	1.0695	9.4	23.4	1.0986	12.9
4.0	1.0157	2.2	10.5	1.0422	5.8	17.0	1.0700	9.4	23.5	1.0991	13.0
4.1	1.0161	2.3	10.6	1.0426	5.9	17.1	1.0704	9.5	23.6	1.0996	13.0
4.2	1.0165	2.3	10.7	1.0430	5.9	17.2	1.0709	9.5	23.7	1.1000	13.1
4.3	1.0169	2.4	10.8	1.0434	6.0	17.3	1.0713	9.6	23.8	1.1005	13.15
4.4	1.0173	2.4	10.9	1.0439	6.05	17.4	1.0717	9.6	23.9	1.1009	13.2
4.5	1.0177	2.5	11.0	1.0443	6.1	17.5	1.0722	9.7	24.0	1.1014	13.3
4.6	1.0181	2.6	11.1	1.0447	6.2	17.6	1.0726	9.75	24.1	1.1019	13.3
4.7	1.0185	2.6	11.2	1.0451	6.2	17.7	1.0730	9.8	24.2	1.1023	13.4
4.8	1.0189	2.7	11.3	1.0455	6.3	17.8	1.0735	9.9	24.3	1.1028	13.4
4.9	1.0193	2.7	11.4	1.0459	6.3	17.9	1.0739	9.9	24.4	1.1032	13.5
5.0	1.0197	2.8	11.5	1.0464	6.4	18.0	1.0744	10.0	24.5	1.1037	13.5
5.1	1.0201	2.8	11.6	1.0468	6.4	18.1	1.0748	10.0	24.6	1.1042	13.6
5.2	1.0205	2.9	11.7	1.0472	6.5	18.2	1.0753	10.1	24.7	1.1046	13.6
5.3	1.0209	2.9	11.8	1.0476	6.55	18.3	1.0757	10.1	24.8	1.1051	13.7
5.4	1.0213	3.0	11.9	1.0481	6.6	18.4	1.0761	10.2	24.9	1.1056	13.75
5.5	1.0217	3.0	12.0	1.0485	6.7	18.5	1.0766	10.2	25.0	1.1060	13.8
5.6	1.0221	3.1	12.1	1.0489	6.7	18.6	1.0770	10.3	25.1	1.1065	13.9
5.7	1.0225	3.2	12.2	1.0493	6.8	18.7	1.0775	10.35	25.2	1.1070	13.9
5.8	1.0229	3.2	12.3	1.0497	6.8	18.8	1.0779	10.4	25.3	1.1074	14.0
5.9	1.0233	3.3	12.4	1.0502	6.9	18.9	1.0783	10.5	25.4	1.1079	14.0
6.0	1.0237	3.3	12.5	1.0506	6.9	19.0	1.0788	10.5	25.5	1.1083	14.1
6.1	1.0241	3.4	12.6	1.0510	7.0	19.1	1.0792	10.6	25.6	1.1088	14.1
6.2	1.0245	3.4	12.7	1.0514	7.05	19.2	1.0797	10.6	25.7	1.1093	14.2
6.3	1.0249	3.5	12.8	1.0519	7.1	19.3	1.0801	10.7	25.8	1.1097	14.2
6.4	1.0253	3.6	12.9	1.0523	7.2	19.4	1.0806	10.7	25.9	1.1102	14.3
6.5	1.0257	3.6	13.0	1.0527	7.2	19.5	1.0810	10.8	26.0	1.1107	14.35

TABLE VI.—*Relation of Brix, specific gravity, and Baumé—Continued.*

Per cent of sugar.	Specific gravity.	Degree Baumé.	Per cent of sugar.	Specific gravity.	Degree Baumé.	Per cent of sugar.	Specific gravity.	Degree Baumé.	Per cent of sugar.	Specific gravity.	Degree Baumé.
26.1	1.1111	14.4	32.6	1.1422	17.9	39.1	1.1748	21.4	45.6	1.2088	24.9
26.2	1.1116	14.5	32.7	1.1427	18.0	39.2	1.1753	21.5	45.7	1.2093	24.9
26.3	1.1121	14.5	32.8	1.1432	18.0	39.3	1.1758	21.5	45.8	1.2099	25.0
26.4	1.1125	14.6	32.9	1.1437	18.1	39.4	1.1763	21.6	45.9	1.2104	25.0
26.5	1.1130	14.6	33.0	1.1442	18.15	39.5	1.1768	21.6	46.0	1.2110	25.1
26.6	1.1135	14.7	33.1	1.1447	18.2	39.6	1.1773	21.7	46.1	1.2115	25.1
26.7	1.1140	14.7	33.2	1.1452	18.25	39.7	1.1778	21.7	46.2	1.2120	25.2
26.8	1.1144	14.8	33.3	1.1457	18.3	39.8	1.1784	21.8	46.3	1.2126	25.2
26.9	1.1149	14.8	33.4	1.1462	18.4	39.9	1.1789	21.85	46.4	1.2131	25.3
27.0	1.1154	14.9	33.5	1.1466	18.4	40.0	1.1794	21.9	46.5	1.2136	25.35
27.1	1.1158	14.9	33.6	1.1471	18.5	40.1	1.1799	22.0	46.6	1.2142	25.4
27.2	1.1163	15.0	33.7	1.1476	18.5	40.2	1.1804	22.0	46.7	1.2147	25.45
27.3	1.1168	15.1	33.8	1.1481	18.6	40.3	1.1809	22.1	46.8	1.2153	25.5
27.4	1.1172	15.1	33.9	1.1486	18.6	40.4	1.1815	22.1	46.9	1.2158	25.6
27.5	1.1177	15.2	34.0	1.1491	18.7	40.5	1.1820	22.2	47.0	1.2163	25.6
27.6	1.1182	15.2	34.1	1.1496	18.7	40.6	1.1825	22.2	47.1	1.2169	25.7
27.7	1.1187	15.3	34.2	1.1501	18.8	40.7	1.1830	22.3	47.2	1.2174	25.7
27.8	1.1191	15.3	34.3	1.1506	18.85	40.8	1.1835	22.3	47.3	1.2180	25.8
27.9	1.1196	15.4	34.4	1.1511	18.9	40.9	1.1840	22.4	47.4	1.2185	25.8
28.0	1.1201	15.4	34.5	1.1516	18.95	41.0	1.1846	22.4	47.5	1.2191	25.9
28.1	1.1206	15.5	34.6	1.1521	19.0	41.1	1.1851	22.5	47.6	1.2196	25.9
28.2	1.1210	15.55	34.7	1.1526	19.1	41.2	1.1856	22.5	47.7	1.2201	26.0
28.3	1.1215	15.6	34.8	1.1531	19.1	41.3	1.1861	22.6	47.8	1.2207	26.0
28.4	1.1220	15.7	34.9	1.1536	19.2	41.4	1.1866	22.65	47.9	1.2212	26.1
28.5	1.1225	15.7	35.0	1.1541	19.2	41.5	1.1872	22.7	48.0	1.2218	26.1
28.6	1.1229	15.8	35.1	1.1546	19.3	41.6	1.1877	22.75	48.1	1.2223	26.2
28.7	1.1234	15.8	35.2	1.1551	19.3	41.7	1.1882	22.8	48.2	1.2229	26.2
28.8	1.1239	15.9	35.3	1.1556	19.4	41.8	1.1887	22.9	48.3	1.2234	26.3
28.9	1.1244	15.9	35.4	1.1561	19.4	41.9	1.1892	22.9	48.4	1.2240	26.35
29.0	1.1248	16.0	35.5	1.1566	19.5	42.0	1.1898	23.0	48.5	1.2245	26.4
29.1	1.1253	16.0	35.6	1.1571	19.55	42.1	1.1903	23.0	48.6	1.2250	26.45
29.2	1.1258	16.1	35.7	1.1576	19.6	42.2	1.1908	23.1	48.7	1.2256	26.5
29.3	1.1263	16.1	35.8	1.1581	19.65	42.3	1.1913	23.1	48.8	1.2261	26.6
29.4	1.1267	16.2	35.9	1.1586	19.7	42.4	1.1919	23.2	48.9	1.2267	26.6
29.5	1.1272	16.25	36.0	1.1591	19.8	42.5	1.1924	23.2	49.0	1.2272	26.7
29.6	1.1277	16.3	36.1	1.1596	19.8	42.6	1.1929	23.3	49.1	1.2278	26.7
29.7	1.1282	16.4	36.2	1.1601	19.9	42.7	1.1934	23.3	49.2	1.2283	26.8
29.8	1.1287	16.4	36.3	1.1606	19.9	42.8	1.1940	23.4	49.3	1.2289	26.8
29.9	1.1291	16.5	36.4	1.1611	20.0	42.9	1.1945	23.45	49.4	1.2294	26.9
30.0	1.1296	16.5	36.5	1.1616	20.0	43.0	1.1950	23.5	49.5	1.2300	26.9
30.1	1.1301	16.6	36.6	1.1621	20.1	43.1	1.1955	23.55	49.6	1.2305	27.0
30.2	1.1306	16.6	36.7	1.1626	20.1	43.2	1.1961	23.6	49.7	1.2311	27.0
30.3	1.1311	16.7	36.8	1.1631	20.2	43.3	1.1966	23.7	49.8	1.2316	27.1
30.4	1.1315	16.7	36.9	1.1636	20.2	43.4	1.1971	23.7	49.9	1.2322	27.1
30.5	1.1320	16.8	37.0	1.1641	20.3	43.5	1.1976	23.8	50.0	1.2327	27.2
30.6	1.1325	16.85	37.1	1.1646	20.35	43.6	1.1982	23.8	50.1	1.2333	27.2
30.7	1.1330	16.9	37.2	1.1651	20.4	43.7	1.1987	23.9	50.2	1.2338	27.3
30.8	1.1335	17.0	37.3	1.1656	20.5	43.8	1.1992	23.9	50.3	1.2344	27.3
30.9	1.1340	17.0	37.4	1.1661	20.5	43.9	1.1998	24.0	50.4	1.2349	27.4
31.0	1.1344	17.1	37.5	1.1666	20.6	44.0	1.2003	24.0	50.5	1.2355	27.45
31.1	1.1349	17.1	37.6	1.1671	20.6	44.1	1.2008	24.1	50.6	1.2361	27.5
31.2	1.1354	17.2	37.7	1.1676	20.7	44.2	1.2013	24.1	50.7	1.2366	27.55
31.3	1.1359	17.2	37.8	1.1681	20.7	44.3	1.2019	24.2	50.8	1.2372	27.6
31.4	1.1364	17.3	37.9	1.1686	20.8	44.4	1.2024	24.2	50.9	1.2377	27.7
31.5	1.1369	17.3	38.0	1.1692	20.8	44.5	1.2029	24.3	51.0	1.2383	27.7
31.6	1.1374	17.4	38.1	1.1697	20.9	44.6	1.2035	24.35	51.1	1.2388	27.8
31.7	1.1378	17.4	38.2	1.1702	20.9	44.7	1.2040	24.4	51.2	1.2394	27.8
31.8	1.1383	17.5	38.3	1.1707	21.0	44.8	1.2045	24.45	51.3	1.2399	27.9
31.9	1.1388	17.55	38.4	1.1712	21.05	44.9	1.2051	24.5	51.4	1.2405	27.9
32.0	1.1393	17.6	38.5	1.1717	21.1	45.0	1.2056	24.6	51.5	1.2411	28.0
32.1	1.1398	17.7	38.6	1.1722	21.15	45.1	1.2061	24.6	51.6	1.2416	28.0
32.2	1.1403	17.7	38.7	1.1727	21.2	45.2	1.2067	24.7	51.7	1.2422	28.1
32.3	1.1408	17.8	38.8	1.1732	21.3	45.3	1.2072	24.7	51.8	1.2427	28.1
32.4	1.1412	17.8	38.9	1.1737	21.3	45.4	1.2077	24.8	51.9	1.2433	28.2
32.5	1.1417	17.9	39.0	1.1743	21.4	45.5	1.2083	24.8	52.0	1.2439	28.2

TABLE VI.—*Relation of Brix, specific gravity, and Baumé—Continued.*

Per cent of sugar.	Specific gravity.	Degree Baumé.	Per cent of sugar.	Specific gravity.	Degree Baumé.	Per cent of sugar.	Specific gravity.	Degree Baumé.	Per cent of sugar.	Specific gravity.	Degree Baumé.
52.1	1.2444	28.3	58.6	1.2816	31.6	65.1	1.3205	34.95	71.6	1.3610	38.2
52.2	1.2450	28.3	58.7	1.2822	31.7	65.2	1.3211	35.0	71.7	1.3616	38.2
52.3	1.2455	28.4	58.8	1.2828	31.7	65.3	1.3217	35.05	71.8	1.3623	38.2
52.4	1.2461	28.4	58.9	1.2834	31.8	65.4	1.3223	35.1	71.9	1.3629	38.3
52.5	1.2467	28.5	59.0	1.2840	31.85	65.5	1.3229	35.15	72.0	1.3635	38.3
52.6	1.2472	28.5	59.1	1.2845	31.9	65.6	1.3235	35.2	72.1	1.3642	38.4
52.7	1.2478	28.6	59.2	1.2851	31.95	65.7	1.3241	35.25	72.2	1.3648	38.4
52.8	1.2483	28.65	59.3	1.2857	32.0	65.8	1.3247	35.3	72.3	1.3655	38.5
52.9	1.2489	28.7	59.4	1.2863	32.05	65.9	1.3253	35.35	72.4	1.3661	38.5
53.0	1.2495	28.75	59.5	1.2869	32.1	66.0	1.3260	35.4	72.5	1.3667	38.6
53.1	1.2500	28.8	59.6	1.2875	32.15	66.1	1.3266	35.4	72.6	1.3674	38.6
53.2	1.2506	28.85	59.7	1.2881	32.2	66.2	1.3272	35.5	72.7	1.3680	38.7
53.3	1.2512	28.9	59.8	1.2887	32.3	66.3	1.3278	35.5	72.8	1.3687	38.7
53.4	1.2517	28.9	59.9	1.2893	32.3	66.4	1.3285	35.6	72.9	1.3693	38.8
53.5	1.2523	29.0	60.0	1.2898	32.4	66.5	1.3291	35.6	73.0	1.3699	38.8
53.6	1.2529	29.1	60.1	1.2904	32.4	66.6	1.3297	35.7	73.1	1.3705	38.9
53.7	1.2534	29.1	60.2	1.2910	32.5	66.7	1.3303	35.7	73.2	1.3712	38.9
53.8	1.2540	29.2	60.3	1.2916	32.5	66.8	1.3309	35.8	73.3	1.3719	39.0
53.9	1.2546	29.2	60.4	1.2922	32.6	66.9	1.3315	35.8	73.4	1.3725	39.0
54.0	1.2551	29.3	60.5	1.2928	32.6	67.0	1.3322	35.9	73.5	1.3732	39.1
54.1	1.2557	29.3	60.6	1.2934	32.7	67.1	1.3327	35.9	73.6	1.3738	39.1
54.2	1.2563	29.4	60.7	1.2940	32.7	67.2	1.3334	36.0	73.7	1.3745	39.2
54.3	1.2568	29.4	60.8	1.2946	32.8	67.3	1.3340	36.0	73.8	1.3751	39.2
54.4	1.2574	29.5	60.9	1.2952	32.8	67.4	1.3346	36.1	73.9	1.3757	39.3
54.5	1.2580	29.5	61.0	1.2958	32.9	67.5	1.3352	36.1	74.0	1.3764	39.3
54.6	1.2585	29.6	61.1	1.2964	32.9	67.6	1.3359	36.2	74.1	1.3770	39.4
54.7	1.2591	29.6	61.2	1.2970	33.0	67.7	1.3365	36.2	74.2	1.3777	39.4
54.8	1.2597	29.7	61.3	1.2975	33.0	67.8	1.3371	36.3	74.3	1.3783	39.5
54.9	1.2602	29.7	61.4	1.2981	33.1	67.9	1.3377	36.3	74.4	1.3790	39.5
55.0	1.2608	29.8	61.5	1.2987	33.1	68.0	1.3384	36.4	74.5	1.3796	39.6
55.1	1.2614	29.8	61.6	1.2993	33.2	68.1	1.3390	36.4	74.6	1.3803	39.6
55.2	1.2620	29.9	61.7	1.2999	33.2	68.2	1.3396	36.5	74.7	1.3809	39.7
55.3	1.2625	29.9	61.8	1.3005	33.3	68.3	1.3402	36.5	74.8	1.3816	39.7
55.4	1.2631	30.0	61.9	1.3011	33.3	68.4	1.3408	36.6	74.9	1.3822	39.8
55.5	1.2637	30.05	62.0	1.3017	33.4	68.5	1.3415	36.6	75.0	1.3828	39.8
55.6	1.2642	30.1	62.1	1.3023	33.4	68.6	1.3421	36.7	75.1	1.3835	39.9
55.7	1.2648	30.15	62.2	1.3029	33.5	68.7	1.3427	36.7	75.2	1.3842	39.9
55.8	1.2654	30.2	62.3	1.3035	33.5	68.8	1.3433	36.8	75.3	1.3848	40.0
55.9	1.2660	30.25	62.4	1.3041	33.6	68.9	1.3440	36.8	75.4	1.3855	40.0
56.0	1.2665	30.3	62.5	1.3047	33.6	69.0	1.3446	36.9	75.5	1.3861	40.1
56.1	1.2671	30.4	62.6	1.3053	33.7	69.1	1.3452	36.9	75.6	1.3868	40.1
56.2	1.2677	30.4	62.7	1.3059	33.7	69.2	1.3458	37.0	75.7	1.3874	40.2
56.3	1.2683	30.5	62.8	1.3065	33.8	69.3	1.3465	37.0	75.8	1.3880	40.2
56.4	1.2688	30.5	62.9	1.3071	33.8	69.4	1.3471	37.1	75.9	1.3887	40.3
56.5	1.2694	30.6	63.0	1.3077	33.9	69.5	1.3477	37.1	76.0	1.3894	40.3
56.6	1.2700	30.6	63.1	1.3083	33.9	69.6	1.3484	37.2	76.1	1.3900	40.4
56.7	1.2706	30.7	63.2	1.3089	34.0	69.7	1.3490	37.2	76.2	1.3907	40.4
56.8	1.2712	30.7	63.3	1.3095	34.0	69.8	1.3496	37.3	76.3	1.3913	40.5
56.9	1.2717	30.8	63.4	1.3101	34.1	69.9	1.3502	37.3	76.4	1.3920	40.5
57.0	1.2723	30.8	63.5	1.3107	34.1	70.0	1.3509	37.4	76.5	1.3926	40.6
57.1	1.2729	30.9	63.6	1.3113	34.2	70.1	1.3515	37.4	76.6	1.3933	40.6
57.2	1.2735	30.9	63.7	1.3119	34.2	70.2	1.3521	37.5	76.7	1.3940	40.7
57.3	1.2740	31.0	63.8	1.3126	34.3	70.3	1.3528	37.5	76.8	1.3946	40.7
57.4	1.2746	31.0	63.9	1.3132	34.3	70.4	1.3534	37.6	76.9	1.3953	40.8
57.5	1.2752	31.1	64.0	1.3138	34.4	70.5	1.3540	37.6	77.0	1.3959	40.8
57.6	1.2758	31.1	64.1	1.3144	34.4	70.6	1.3546	37.7	77.1	1.3966	40.8
57.7	1.2764	31.2	64.2	1.3150	34.5	70.7	1.3553	37.7	77.2	1.3972	40.9
57.8	1.2769	31.2	64.3	1.3156	34.5	70.8	1.3559	37.8	77.3	1.3979	41.0
57.9	1.2775	31.3	64.4	1.3162	34.6	70.9	1.3565	37.8	77.4	1.3986	41.0
58.0	1.2781	31.3	64.5	1.3168	34.6	71.0	1.3572	37.9	77.5	1.3992	41.0
58.1	1.2787	31.4	64.6	1.3174	34.7	71.1	1.3578	37.9	77.6	1.3999	41.1
58.2	1.2793	31.4	64.7	1.3180	34.7	71.2	1.3585	38.0	77.7	1.4005	41.1
58.3	1.2799	31.5	64.8	1.3186	34.8	71.3	1.3591	38.0	77.8	1.4012	41.2
58.4	1.2804	31.5	64.9	1.3192	34.8	71.4	1.3597	38.1	77.9	1.4019	41.2
58.5	1.2810	31.6	65.0	1.3198	34.9	71.5	1.3604	38.1	78.0	1.4025	41.3

TABLE VI.—*Relation of Brix, specific gravity, and Baumé—Continued.*

Per cent of sugar	Specific gravity.	Degree Baumé.	Per cent of sugar.	Specific gravity.	Degree Baumé.	Per cent of sugar.	Specific gravity.	Degree Baumé.	Per cent of sugar.	Specific gravity.	Degree Baumé.
78.1	1.4032	41.3	80.1	1.4165	42.3	82.1	1.4300	43.3	84.1	1.4437	44.2
78.2	1.4039	41.4	80.2	1.4172	42.3	82.2	1.4307	43.3	84.2	1.4443	44.3
78.3	1.4045	41.4	80.3	1.4179	42.4	82.3	1.4314	43.4	84.3	1.4450	44.3
78.4	1.4052	41.5	80.4	1.4185	42.4	82.4	1.4320	43.4	84.4	1.4457	44.3
78.5	1.4058	41.5	80.5	1.4192	42.5	82.5	1.4327	43.5	84.5	1.4464	44.4
78.6	1.4065	41.6	80.6	1.4199	42.5	82.6	1.4334	43.5	84.6	1.4471	44.4
78.7	1.4072	41.6	80.7	1.4205	42.6	82.7	1.4341	43.5	84.7	1.4478	44.5
78.8	1.4078	41.7	80.8	1.4212	42.6	82.8	1.4348	43.6	84.8	1.4485	44.5
78.9	1.4085	41.7	80.9	1.4219	42.7	82.9	1.4354	43.6	84.9	1.4492	44.6
79.0	1.4092	41.8	81.0	1.4226	42.7	83.0	1.4361	43.7	85.0	1.4498	44.6
79.1	1.4098	41.8	81.1	1.4232	42.8	83.1	1.4368	43.7	85.1	1.4505	44.7
79.2	1.4105	41.9	81.2	1.4239	42.8	83.2	1.4375	43.8	85.2	1.4512	44.7
79.3	1.4112	41.9	81.3	1.4246	42.9	83.3	1.4382	43.8	85.3	1.4519	44.8
79.4	1.4119	42.0	81.4	1.4253	42.9	83.4	1.4388	43.9	85.4	1.4526	44.8
79.5	1.4125	42.0	81.5	1.4259	43.0	83.5	1.4395	43.9	85.5	1.4533	44.9
79.6	1.4132	42.1	81.6	1.4266	43.0	83.6	1.4402	44.0	85.6	1.4540	44.9
79.7	1.4138	42.1	81.7	1.4273	43.1	83.7	1.4409	44.0	85.7	1.4547	45.0
79.8	1.4145	42.2	81.8	1.4280	43.1	83.8	1.4416	44.1	85.8	1.4554	45.0
79.9	1.4152	42.2	81.9	1.4287	43.2	83.9	1.4423	44.1	85.9	1.4561	45.1
80.0	1.4158	42.2	82.0	1.4293	43.2	84.0	1.4430	44.2	86.0	1.4568	45.1

TABLE VII.—*Correction for the readings of Balling's saccharometer, on account of temperature.*

TO BE SUBTRACTED FROM THE DEGREE READ.

Temp.	Per cent of sugar in solution.												
C.	0	5	10	15	20	25	30	35	40	50	60	70	75
13	0.14	0.18	0.19	0.21	0.22	0.24	0.26	0.27	0.28	0.29	0.33	0.35	0.39
14	.12	.15	.16	.17	.18	.19	.21	.22	.22	.23	.26	.28	.32
15	.09	.11	.12	.14	.14	.15	.16	.17	.16	.17	.19	.21	.25
16	.06	.07	.08	.09	.10	.10	.11	.12	.12	.12	.14	.16	.18
17	.02	.02	.03	.03	.03	.04	.04	.04	.04	.04	.05	.05	.06
TO BE ADDED TO THE DEGREE READ.													
18	.02	.03	.03	.03	.03	.03	.03	.03	.03	.03	.03	.03	.02
19	.06	.08	.08	.09	.09	.10	.10	.10	.10	.10	.10	.08	.06
20	.11	.14	.15	.17	.17	.18	.18	.18	.19	.19	.18	.15	.11
21	.16	.20	.22	.24	.24	.25	.25	.25	.26	.26	.25	.22	.18
22	.21	.26	.29	.31	.31	.32	.32	.32	.33	.34	.32	.29	.25
23	.27	.32	.35	.37	.38	.39	.39	.39	.40	.42	.39	.36	.33
24	.32	.38	.41	.43	.44	.46	.46	.47	.47	.50	.46	.43	.40
25	.37	.44	.47	.49	.51	.53	.54	.55	.55	.58	.54	.51	.48
26	.43	.50	.54	.56	.58	.60	.61	.62	.62	.66	.62	.58	.55
27	.49	.57	.61	.63	.65	.68	.68	.69	.70	.74	.70	.65	.62
28	.56	.64	.68	.70	.72	.76	.76	.78	.78	.82	.78	.72	.70
29	.63	.71	.75	.78	.79	.84	.84	.86	.86	.90	.86	.80	.78
30	.70	.78	.82	.87	.87	.92	.92	.94	.94	.98	.94	.88	.86



TABLE VIII.—*Per cent of fat and solids not fat in milk.*

[According to Babcock.]

Per cent of fat.	Lactometer readings at 15.6° C.											Per cent of fat.
	26.	27.	28.	29.	30.	31.	32.	33.	34.	35.	36.	
0.0	6.50	6.75	7.00	7.25	7.50	7.75	8.00	8.25	8.50	8.75	9.00	0.0
0.1	6.52	6.77	7.02	7.27	7.52	7.77	8.02	8.27	8.52	8.77	9.02	0.1
0.2	6.24	6.79	7.04	7.29	7.54	7.79	8.04	8.29	8.54	8.79	9.04	0.2
0.3	6.56	6.81	7.06	7.31	7.56	7.81	8.06	8.31	8.56	8.81	9.06	0.3
0.4	6.58	6.83	7.08	7.33	7.58	7.83	8.08	8.33	8.58	8.83	9.08	0.4
0.5	6.60	6.85	7.10	7.35	7.60	7.85	8.10	8.35	8.60	8.85	9.10	0.5
0.6	6.62	6.87	7.12	7.37	7.62	7.87	8.12	8.37	8.62	8.87	9.12	0.6
0.7	6.64	6.89	7.14	7.39	7.64	7.89	8.14	8.39	8.64	8.89	9.14	0.7
0.8	6.66	6.91	7.16	7.41	7.66	7.91	8.16	8.41	8.66	8.91	9.16	0.8
0.9	6.68	6.93	7.18	7.43	7.68	7.93	8.18	8.43	8.68	8.93	9.18	0.9
1.0	6.70	6.95	7.20	7.45	7.70	7.95	8.20	8.45	8.70	8.95	9.20	1.0
1.1	6.72	6.97	7.22	7.47	7.72	7.97	8.22	8.47	8.72	8.97	9.22	1.1
1.2	6.74	6.99	7.24	7.49	7.74	7.99	8.24	8.49	8.74	8.99	9.24	1.2
1.3	6.76	7.01	7.26	7.51	7.76	8.01	8.26	8.51	8.76	9.01	9.26	1.3
1.4	6.78	7.03	7.28	7.53	7.78	8.03	8.28	8.53	8.78	9.03	9.28	1.4
1.5	6.80	7.05	7.30	7.55	7.80	8.05	8.30	8.55	8.80	9.05	9.30	1.5
1.6	6.82	7.07	7.32	7.57	7.82	8.07	8.32	8.57	8.82	9.07	9.32	1.6
1.7	6.84	7.09	7.34	7.59	7.84	8.09	8.34	8.59	8.84	9.09	9.34	1.7
1.8	6.86	7.11	7.36	7.61	7.86	8.11	8.36	8.61	8.86	9.11	9.37	1.8
1.9	6.88	7.13	7.38	7.63	7.88	8.13	8.38	8.63	8.88	9.13	9.39	1.9
2.0	6.90	7.15	7.40	7.65	7.90	8.15	8.40	8.66	8.91	9.16	9.41	2.0
2.1	6.92	7.17	7.42	7.67	7.92	8.17	8.42	8.68	8.93	9.18	9.43	2.1
2.2	6.94	7.19	7.44	7.69	7.94	8.19	8.44	8.70	8.95	9.20	9.45	2.2
2.3	6.96	7.21	7.46	7.71	7.96	8.21	8.46	8.72	8.97	9.22	9.47	2.3
2.4	6.98	7.23	7.48	7.73	7.98	8.23	8.48	8.74	8.99	9.24	9.49	2.4
2.5	7.00	7.25	7.50	7.75	8.00	8.25	8.50	8.76	9.01	9.26	9.51	2.5
2.6	7.02	7.27	7.52	7.77	8.02	8.27	8.52	8.78	9.03	9.28	9.53	2.6
2.7	7.04	7.29	7.54	7.79	8.04	8.29	8.54	8.80	9.05	9.30	9.55	2.7
2.8	7.06	7.31	7.56	7.81	8.06	8.31	8.57	8.82	9.07	9.32	9.57	2.8
2.9	7.08	7.33	7.58	7.83	8.08	8.33	8.59	8.84	9.09	9.34	9.59	2.9
3.0	7.10	7.35	7.60	7.85	8.10	8.36	8.61	8.86	9.11	9.36	9.61	3.0
3.1	7.12	7.37	7.62	7.87	8.13	8.38	8.63	8.88	9.13	9.38	9.64	3.1
3.2	7.14	7.39	7.64	7.89	8.15	8.40	8.65	8.90	9.15	9.41	9.66	3.2
3.3	7.16	7.41	7.66	7.92	8.17	8.42	8.67	8.92	9.18	9.43	9.68	3.3
3.4	7.18	7.43	7.69	7.94	8.19	8.44	8.69	8.94	9.20	9.45	9.70	3.4
3.5	7.20	7.45	7.71	7.96	8.21	8.46	8.71	8.96	9.22	9.47	9.72	3.5
3.6	7.22	7.48	7.73	7.98	8.23	8.48	8.73	8.98	9.24	9.49	9.74	3.6
3.7	7.24	7.50	7.75	8.00	8.25	8.50	8.75	9.00	9.26	9.51	9.76	3.7
3.8	7.26	7.52	7.77	8.02	8.27	8.52	8.77	9.02	9.28	9.53	9.78	3.8
3.9	7.28	7.54	7.79	8.04	8.29	8.54	8.79	9.04	9.30	9.55	9.80	3.9
4.0	7.30	7.56	7.81	8.06	8.31	8.56	8.81	9.06	9.32	9.57	9.83	4.0
4.1	7.32	7.58	7.83	8.08	8.33	8.58	8.83	9.08	9.34	9.59	9.85	4.1
4.2	7.34	7.60	7.85	8.10	8.35	8.60	8.85	9.11	9.36	9.62	9.87	4.2
4.3	7.36	7.62	7.87	8.12	8.37	8.62	8.88	9.13	9.38	9.64	9.89	4.3
4.4	7.38	7.64	7.89	8.14	8.39	8.64	8.90	9.15	9.40	9.66	9.91	4.4
4.5	7.40	7.66	7.91	8.16	8.41	8.66	8.92	9.17	9.42	9.68	9.93	4.5
4.6	7.43	7.68	7.93	8.18	8.43	8.68	8.94	9.19	9.44	9.70	9.95	4.6
4.7	7.45	7.70	7.95	8.20	8.45	8.70	8.96	9.21	9.46	9.72	9.97	4.7
4.8	7.47	7.72	7.97	8.22	8.47	8.72	8.98	9.23	9.48	9.74	9.99	4.8
4.9	7.49	7.74	7.99	8.24	8.49	8.74	9.00	9.25	9.50	9.76	10.01	4.9
5.0	7.51	7.76	8.01	8.26	8.51	8.76	9.02	9.27	9.52	9.78	10.03	5.0
5.1	7.53	7.78	8.03	8.28	8.53	8.79	9.04	9.29	9.54	9.80	10.05	5.1
5.2	7.55	7.80	8.05	8.30	8.55	8.81	9.06	9.31	9.56	9.82	10.07	5.2
5.3	7.57	7.82	8.07	8.32	8.57	8.83	9.08	9.33	9.58	9.84	10.09	5.3
5.4	7.59	7.84	8.09	8.34	8.60	8.85	9.10	9.36	9.61	9.86	10.11	5.4
5.5	7.61	7.86	8.11	8.36	8.62	8.87	9.12	9.38	9.63	9.88	10.13	5.5
5.6	7.63	7.88	8.13	8.39	8.64	8.89	9.15	9.40	9.65	9.90	10.15	5.6
5.7	7.65	7.90	8.15	8.41	8.66	8.91	9.17	9.42	9.67	9.92	10.17	5.7
5.8	7.67	7.92	8.17	8.43	8.68	8.94	9.19	9.44	9.69	9.94	10.19	5.8
5.9	7.69	7.94	8.20	8.45	8.70	8.96	9.21	9.46	9.71	9.96	10.22	5.9
6.0	7.71	7.96	8.22	8.47	8.72	8.98	9.23	9.48	9.73	9.98	10.24	6.0

TABLE IX.—*Determination of pentoses and pentosans from phloroglucid (Kröber).<sup>a</sup>*

1. Phloro- glucid.	2. Furfural.	3. Arabi- nose.	4. Araban.	5. Xylose.	6. Xylan.	7. Pentose.	8. Pentosan.
0.030	0.0182	0.0391	0.0344	0.0324	0.0285	0.0358	0.0315
.031	.0188	.0402	.0354	.0333	.0293	.0368	.0324
.032	.0193	.0413	.0363	.0342	.0301	.0378	.0333
.033	.0198	.0424	.0373	.0352	.0309	.0388	.0341
.034	.0203	.0435	.0383	.0361	.0317	.0398	.0350
.035	.0209	.0446	.0393	.0370	.0326	.0408	.0359
.036	.0214	.0457	.0402	.0379	.0334	.0418	.0368
.037	.0219	.0468	.0412	.0388	.0342	.0428	.0377
.038	.0224	.0479	.0422	.0398	.0350	.0439	.0386
.039	.0229	.0490	.0431	.0407	.0358	.0449	.0395
.040	.0235	.0501	.0441	.0416	.0366	.0459	.0404
.041	.0240	.0512	.0451	.0425	.0374	.0469	.0413
.042	.0245	.0523	.0460	.0434	.0382	.0479	.0422
.043	.0250	.0534	.0470	.0443	.0390	.0489	.0431
.044	.0255	.0545	.0480	.0452	.0398	.0499	.0440
.045	.0260	.0556	.0490	.0462	.0406	.0509	.0448
.046	.0266	.0567	.0499	.0471	.0414	.0519	.0457
.047	.0271	.0578	.0509	.0480	.0422	.0529	.0466
.048	.0276	.0589	.0519	.0489	.0430	.0539	.0475
.049	.0281	.0600	.0528	.0498	.0438	.0549	.0484
.050	.0286	.0611	.0538	.0507	.0446	.0559	.0492
.051	.0292	.0622	.0548	.0516	.0454	.0569	.0501
.052	.0297	.0633	.0557	.0525	.0462	.0579	.0510
.053	.0302	.0644	.0567	.0534	.0470	.0589	.0519
.054	.0307	.0655	.0576	.0543	.0478	.0599	.0528
.055	.0312	.0666	.0586	.0553	.0486	.0610	.0537
.056	.0318	.0677	.0596	.0562	.0494	.0620	.0546
.057	.0323	.0688	.0605	.0571	.0502	.0630	.0555
.058	.0328	.0699	.0615	.0580	.0510	.0640	.0564
.059	.0333	.0710	.0624	.0589	.0518	.0650	.0573
.060	.0338	.0721	.0634	.0598	.0526	.0660	.0581
.061	.0344	.0732	.0644	.0607	.0534	.0670	.0590
.062	.0349	.0743	.0653	.0616	.0542	.0680	.0599
.063	.0354	.0754	.0663	.0626	.0550	.0690	.0608
.064	.0359	.0765	.0673	.0635	.0558	.0700	.0617
.065	.0364	.0776	.0683	.0644	.0567	.0710	.0625
.066	.0370	.0787	.0692	.0653	.0575	.0720	.0634
.067	.0375	.0798	.0702	.0662	.0583	.0730	.0643
.068	.0380	.0809	.0712	.0672	.0591	.0741	.0652
.069	.0385	.0820	.0721	.0681	.0599	.0751	.0661
.070	.0390	.0831	.0731	.0690	.0607	.0761	.0670
.071	.0396	.0842	.0741	.0699	.0615	.0771	.0679
.072	.0401	.0853	.0750	.0708	.0623	.0781	.0688
.073	.0406	.0864	.0760	.0717	.0631	.0791	.0697
.074	.0411	.0875	.0770	.0726	.0639	.0801	.0706
.075	.0416	.0886	.0780	.0736	.0647	.0811	.0714
.076	.0422	.0897	.0789	.0745	.0655	.0821	.0722
.077	.0427	.0908	.0799	.0754	.0663	.0831	.0731
.078	.0432	.0919	.0809	.0763	.0671	.0841	.0740
.079	.0437	.0930	.0818	.0772	.0679	.0851	.0749
.080	.0442	.0941	.0828	.0781	.0687	.0861	.0758
.081	.0448	.0952	.0838	.0790	.0695	.0871	.0767
.082	.0453	.0963	.0847	.0799	.0703	.0881	.0776
.083	.0458	.0974	.0857	.0808	.0711	.0891	.0785
.084	.0463	.0985	.0867	.0817	.0719	.0901	.0794
.085	.0468	.0996	.0877	.0827	.0727	.0912	.0803
.086	.0474	.1007	.0886	.0836	.0735	.0922	.0812
.087	.0479	.1018	.0896	.0845	.0743	.0932	.0821
.088	.0484	.1029	.0906	.0854	.0751	.0942	.0830
.089	.0489	.1040	.0915	.0863	.0759	.0952	.0838
.090	.0494	.1051	.0925	.0872	.0767	.0962	.0847
.091	.0499	.1062	.0935	.0881	.0775	.0972	.0856
.092	.0505	.1073	.0944	.0890	.0783	.0982	.0865
.093	.0510	.1084	.0954	.0900	.0791	.0992	.0874
.094	.0515	.1095	.0964	.0909	.0800	.1002	.0883

TABLE IX.—*Determination of pentoses and pentosans, etc.—Continued.*

1. Phloro- glucid.	2. Furfural.	3. Arabi- nose.	4. Arab. an.	5. Xylose.	6. Xylan.	7. Pentose.	8. Pentosan.
0.095	0.0520	0.1106	0.0974	0.0918	0.0808	0.1012	0.0891
.096	.0525	.1117	.0983	.0927	.0816	.1022	.0899
.097	.0531	.1128	.0793	.0936	.0824	.1032	.0908
.098	.0536	.1139	.1003	.0946	.0832	.1043	.0917
.099	.0541	.1150	.1012	.0955	.0840	.1053	.0926
.100	.0546	.1161	.1022	.0964	.0848	.1063	.0935
.101	.0551	.1171	.1032	.0973	.0856	.1073	.0944
.102	.0557	.1182	.1041	.0982	.0864	.1083	.0953
.103	.0562	.1193	.1051	.0991	.0872	.1093	.0962
.104	.0567	.1204	.1060	.1000	.0880	.1103	.0971
.105	.0572	.1215	.1070	.1010	.0888	.1113	.0976
.106	.0577	.1226	.1080	.1019	.0896	.1123	.0988
.107	.0582	.1237	.1089	.1028	.0904	.1133	.0997
.108	.0588	.1248	.1099	.1037	.0912	.1143	.1006
.109	.0593	.1259	.1108	.1046	.0920	.1153	.1015
.110	.0598	.1270	.1118	.1055	.0928	.1163	.1023
.111	.0603	.1281	.1128	.1064	.0936	.1173	.1032
.112	.0608	.1292	.1137	.1073	.0944	.1183	.1041
.113	.0614	.1303	.1147	.1082	.0952	.1193	.1050
.114	.0619	.1314	.1156	.1091	.0960	.1203	.1059
.115	.0624	.1325	.1166	.1101	.0968	.1213	.1067
.116	.0629	.1336	.1176	.1110	.0976	.1223	.1076
.117	.0634	.1347	.1185	.1119	.0984	.1233	.1085
.118	.0640	.1358	.1195	.1128	.0992	.1243	.1094
.119	.0645	.1369	.1204	.1137	.1000	.1253	.1103
.120	.0650	.1380	.1214	.1146	.1008	.1263	.1111
.121	.0655	.1391	.1224	.1155	.1016	.1273	.1120
.122	.0660	.1402	.1233	.1164	.1024	.1283	.1129
.123	.0665	.1413	.1243	.1173	.1032	.1293	.1138
.124	.0671	.1424	.1253	.1182	.1040	.1303	.1147
.125	.0676	.1435	.1263	.1192	.1049	.1314	.1156
.126	.0681	.1446	.1272	.1201	.1057	.1324	.1165
.127	.0686	.1457	.1282	.1210	.1065	.1334	.1174
.128	.0691	.1468	.1292	.1219	.1073	.1344	.1183
.129	.0697	.1479	.1301	.1228	.1081	.1354	.1192
.130	.0702	.1490	.1311	.1237	.1089	.1364	.1201
.131	.0707	.1501	.1321	.1246	.1097	.1374	.1210
.132	.0712	.1512	.1330	.1255	.1105	.1384	.1219
.133	.0717	.1523	.1340	.1264	.1113	.1394	.1227
.134	.0723	.1534	.1350	.1273	.1121	.1404	.1236
.135	.0728	.1545	.1360	.1283	.1129	.1414	.1244
.136	.0733	.1556	.1369	.1292	.1137	.1424	.1253
.137	.0738	.1567	.1379	.1301	.1145	.1434	.1262
.138	.0743	.1578	.1389	.1310	.1153	.1444	.1271
.139	.0748	.1589	.1398	.1319	.1161	.1454	.1280
.140	.0754	.1600	.1408	.1328	.1169	.1464	.1288
.141	.0759	.1611	.1418	.1337	.1177	.1474	.1297
.142	.0764	.1622	.1427	.1346	.1185	.1484	.1306
.143	.0769	.1633	.1437	.1355	.1193	.1494	.1315
.144	.0774	.1644	.1447	.1364	.1201	.1504	.1324
.145	.0780	.1655	.1457	.1374	.1209	.1515	.1333
.146	.0785	.1666	.1466	.1383	.1217	.1525	.1342
.147	.0790	.1677	.1476	.1392	.1225	.1535	.1351
.148	.0795	.1688	.1486	.1401	.1233	.1545	.1360
.149	.0800	.1699	.1495	.1410	.1241	.1555	.1369
.150	.0805	.1710	.1505	.1419	.1249	.1565	.1377
.151	.0811	.1721	.1515	.1428	.1257	.1575	.1386
.152	.0816	.1732	.1524	.1437	.1265	.1585	.1395
.153	.0821	.1743	.1534	.1446	.1273	.1595	.1404
.154	.0826	.1754	.1544	.1455	.1281	.1605	.1413
.155	.0831	.1765	.1554	.1465	.1289	.1615	.1421
.156	.0837	.1776	.1563	.1474	.1297	.1625	.1430
.157	.0842	.1787	.1573	.1483	.1305	.1635	.1439
.158	.0847	.1798	.1583	.1492	.1313	.1645	.1448
.159	.0852	.1809	.1592	.1501	.1321	.1655	.1457

TABLE IX.—*Determination of pentoses and pentosans, etc.*—Continued.

1. Phloro- glucid.	2. Furfural.	3. Arabi- nose.	4. Arabian.	5. Xylose.	6. Xylan.	7. Pentose.	8. Pentosan.
0.160	0.0857	0.1820	0.1602	0.1510	0.1329	0.1665	0.1465
.161	.0863	.1831	.1612	.1519	.1337	.1675	.1474
.162	.0868	.1842	.1621	.1528	.1345	.1685	.1483
.163	.0873	.1853	.1631	.1537	.1353	.1695	.1492
.164	.0878	.1864	.1640	.1546	.1361	.1705	.1501
.165	.0883	.1875	.1650	.1556	.1369	.1716	.1510
.166	.0888	.1886	.1660	.1565	.1377	.1726	.1519
.167	.0894	.1897	.1669	.1574	.1385	.1736	.1528
.168	.0899	.1908	.1679	.1583	.1393	.1746	.1537
.169	.0904	.1919	.1688	.1592	.1401	.1756	.1546
.170	.0909	.1930	.1698	.1601	.1409	.1766	.1554
.171	.0914	.1941	.1708	.1610	.1417	.1776	.1563
.172	.0920	.1952	.1717	.1619	.1425	.1786	.1572
.173	.0925	.1963	.1727	.1628	.1433	.1796	.1581
.174	.0930	.1974	.1736	.1637	.1441	.1806	.1590
.175	.0935	.1985	.1746	.1647	.1449	.1816	.1598
.176	.0940	.1996	.1756	.1656	.1457	.1826	.1607
.177	.0946	.2007	.1765	.1665	.1465	.1836	.1616
.178	.0951	.2018	.1775	.1674	.1473	.1846	.1625
.179	.0956	.2029	.1784	.1683	.1481	.1856	.1634
.180	.0961	.2039	.1794	.1692	.1489	.1866	.1642
.181	.0966	.2050	.1804	.1701	.1497	.1876	.1651
.182	.0971	.2061	.1813	.1710	.1505	.1886	.1660
.183	.0977	.2072	.1823	.1719	.1513	.1896	.1669
.184	.0982	.2082	.1832	.1728	.1521	.1906	.1678
.185	.0987	.2093	.1842	.1738	.1529	.1916	.1686
.186	.0992	.2104	.1851	.1747	.1537	.1926	.1695
.187	.0997	.2115	.1861	.1756	.1545	.1936	.1704
.188	.1003	.2126	.1870	.1765	.1553	.1946	.1712
.189	.1008	.2136	.1880	.1774	.1561	.1955	.1721
.190	.1013	.2147	.1889	.1783	.1569	.1965	.1729
.191	.1018	.2158	.1899	.1792	.1577	.1975	.1738
.192	.1023	.2168	.1908	.1801	.1585	.1985	.1747
.193	.1028	.2179	.1918	.1810	.1593	.1995	.1756
.194	.1034	.2190	.1927	.1819	.1601	.2005	.1764
.195	.1039	.2201	.1937	.1829	.1609	.2015	.1773
.196	.1044	.2212	.1946	.1838	.1617	.2025	.1782
.197	.1049	.2222	.1956	.1847	.1625	.2035	.1791
.198	.1054	.2233	.1965	.1856	.1633	.2045	.1800
.199	.1059	.2244	.1975	.1865	.1641	.2055	.1808
.200	.1065	.2255	.1984	.1874	.1649	.2065	.1817
.201	.1070	.2266	.1994	.1883	.1657	.2075	.1826
.202	.1075	.2276	.2003	.1892	.1665	.2085	.1835
.203	.1080	.2287	.2013	.1901	.1673	.2095	.1844
.204	.1085	.2298	.2022	.1910	.1681	.2105	.1853
.205	.1090	.2309	.2032	.1920	.1689	.2115	.1861
.206	.1096	.2320	.2041	.1929	.1697	.2125	.1869
.207	.1101	.2330	.2051	.1938	.1705	.2134	.1878
.208	.1106	.2341	.2060	.1947	.1713	.2144	.1887
.209	.1111	.2352	.2069	.1956	.1721	.2154	.1896
.210	.1116	.2363	.2079	.1965	.1729	.2164	.1904
.211	.1121	.2374	.2089	.1975	.1737	.2174	.1913
.212	.1127	.2384	.2098	.1984	.1745	.2184	.1922
.213	.1132	.2395	.2108	.1993	.1753	.2194	.1931
.214	.1137	.2406	.2117	.2002	.1761	.2204	.1940
.215	.1142	.2417	.2127	.2011	.1770	.2214	.1948
.216	.1147	.2428	.2136	.2020	.1778	.2224	.1957
.217	.1152	.2438	.2146	.2029	.1786	.2234	.1966
.218	.1158	.2449	.2155	.2038	.1794	.2244	.1974
.219	.1163	.2460	.2165	.2047	.1802	.2254	.1983
.220	.1168	.2471	.2174	.2057	.1810	.2264	.1992
.221	.1173	.2482	.2184	.2066	.1818	.2274	.2001
.222	.1178	.2492	.2193	.2075	.1826	.2284	.2010
.223	.1183	.2503	.2203	.2084	.1834	.2294	.2019
.224	.1189	.2514	.2212	.2093	.1842	.2304	.2028

TABLE IX.—*Determination of pentoses and pentosans, etc.* Continued.

1. Phloro- glucid.	2. Furfural.	3. Arabi- nose.	4. Araban.	5. Xylose.	6. Xylan.	7. Pentose.	8. Pentosan.
0.225	0.1194	0.2525	0.2222	0.2102	0.1850	0.2314	0.2037
.226	.1199	.2536	.2232	.2111	.1858	.2324	.2046
.227	.1204	.2546	.2241	.2121	.1866	.2334	.2054
.228	.1209	.2557	.2251	.2130	.1874	.2344	.2063
.229	.1214	.2568	.2260	.2139	.1882	.2354	.2072
.230	.1220	.2579	.2270	.2148	.1890	.2364	.2081
.231	.1225	.2590	.2280	.2157	.1898	.2374	.2089
.232	.1230	.2600	.2289	.2166	.1906	.2383	.2097
.233	.1235	.2611	.2299	.2175	.1914	.2393	.2106
.234	.1240	.2622	.2308	.2184	.1922	.2403	.2115
.235	.1245	.2633	.2318	.2193	.1930	.2413	.2124
.236	.1251	.2644	.2327	.2202	.1938	.2423	.2132
.237	.1256	.2654	.2337	.2211	.1946	.2433	.2141
.238	.1261	.2665	.2346	.2220	.1954	.2443	.2150
.239	.1266	.2676	.2356	.2229	.1962	.2453	.2159
.240	.1271	.2687	.2365	.2239	.1970	.2463	.2168
.241	.1276	.2698	.2375	.2248	.1978	.2473	.2176
.242	.1281	.2708	.2384	.2257	.1986	.2483	.2185
.243	.1287	.2719	.2394	.2266	.1994	.2493	.2194
.244	.1292	.2730	.2403	.2275	.2002	.2503	.2203
.245	.1297	.2741	.2413	.2284	.2010	.2513	.2212
.246	.1302	.2752	.2422	.2293	.2018	.2523	.2220
.247	.1307	.2762	.2432	.2302	.2026	.2533	.2229
.248	.1312	.2773	.2441	.2311	.2034	.2543	.2238
.249	.1318	.2784	.2451	.2320	.2042	.2553	.2247
.250	.1323	.2795	.2460	.2330	.2050	.2563	.2256
.251	.1328	.2806	.2470	.2339	.2058	.2573	.2264
.252	.1333	.2816	.2479	.2348	.2066	.2582	.2272
.253	.1338	.2827	.2489	.2357	.2074	.2592	.2281
.254	.1343	.2838	.2498	.2366	.2082	.2602	.2290
.255	.1349	.2849	.2508	.2375	.2090	.2612	.2299
.256	.1354	.2860	.2517	.2384	.2098	.2622	.2307
.257	.1359	.2870	.2526	.2393	.2106	.2632	.2316
.258	.1364	.2881	.2536	.2402	.2114	.2642	.2325
.259	.1369	.2892	.2545	.2411	.2122	.2652	.2334
.260	.1374	.2903	.2555	.2420	.2130	.2662	.2343
.261	.1380	.2914	.2565	.2429	.2138	.2672	.2351
.262	.1385	.2924	.2574	.2438	.2146	.2681	.2359
.263	.1390	.2935	.2584	.2447	.2154	.2691	.2368
.264	.1395	.2946	.2593	.2456	.2162	.2701	.2377
.265	.1400	.2957	.2603	.2465	.2170	.2711	.2385
.266	.1405	.2968	.2612	.2474	.2178	.2721	.2394
.267	.1411	.2978	.2622	.2483	.2186	.2731	.2403
.268	.1416	.2989	.2631	.2492	.2194	.2741	.2412
.269	.1421	.3000	.2641	.2502	.2202	.2751	.2421
.270	.1426	.3011	.2650	.2511	.2210	.2761	.2429
.271	.1431	.3022	.2660	.2520	.2218	.2771	.2438
.272	.1436	.3032	.2669	.2529	.2226	.2781	.2447
.273	.1442	.3043	.2679	.2538	.2234	.2791	.2456
.274	.1447	.3054	.2688	.2547	.2242	.2801	.2465
.275	.1452	.3065	.2698	.2556	.2250	.2811	.2473
.276	.1457	.3076	.2707	.2565	.2258	.2821	.2482
.277	.1462	.3086	.2717	.2574	.2266	.2830	.2490
.278	.1467	.3097	.2726	.2583	.2274	.2840	.2499
.279	.1473	.3108	.2736	.2592	.2282	.2850	.2508
.280	.1478	.3119	.2745	.2602	.2290	.2861	.2517
.281	.1483	.3130	.2755	.2611	.2298	.2871	.2526
.282	.1488	.3140	.2764	.2620	.2306	.2880	.2534
.283	.1493	.3151	.2774	.2629	.2314	.2890	.2543
.284	.1498	.3162	.2783	.2638	.2322	.2900	.2552
.285	.1504	.3173	.2793	.2647	.2330	.2910	.2561
.286	.1509	.3184	.2802	.2656	.2338	.2920	.2570
.287	.1514	.3194	.2812	.2665	.2346	.2930	.2578
.288	.1519	.3205	.2821	.2674	.2354	.2940	.2587
.289	.1524	.3216	.2831	.2683	.2362	.2950	.2596

TABLE IX.—*Determination of pentoses and pentosans, etc.*—Continued.

1. Phloro- glucid.	2. Furfural.	3. Arabi- nose.	4. Araban.	5. Xylose.	6. Xylan.	7. Pentose.	8. Pentosan.
0.290	0.1529	0.3227	0.2840	0.2693	0.2370	0.2960	0.2605
.291	.1535	.3238	.2850	.2702	.2378	.2970	.2614
.292	.1540	.3248	.2859	.2711	.2386	.2980	.2622
.293	.1545	.3259	.2868	.2720	.2394	.2990	.2631
.294	.1550	.3270	.2878	.2729	.2402	.3000	.2640
.295	.1555	.3281	.2887	.2738	.2410	.3010	.2649
.296	.1560	.3292	.2897	.2747	.2418	.3020	.2658
.297	.1566	.3302	.2906	.2756	.2426	.3030	.2666
.298	.1571	.3313	.2916	.2765	.2434	.3040	.2675
.299	.1576	.3324	.2925	.2774	.2442	.3050	.2684
.300	.1581	.3335	.2935	.2784	.2450	.3060	.2693

TABLE X.—*International atomic weights, 1907.<sup>a</sup>*

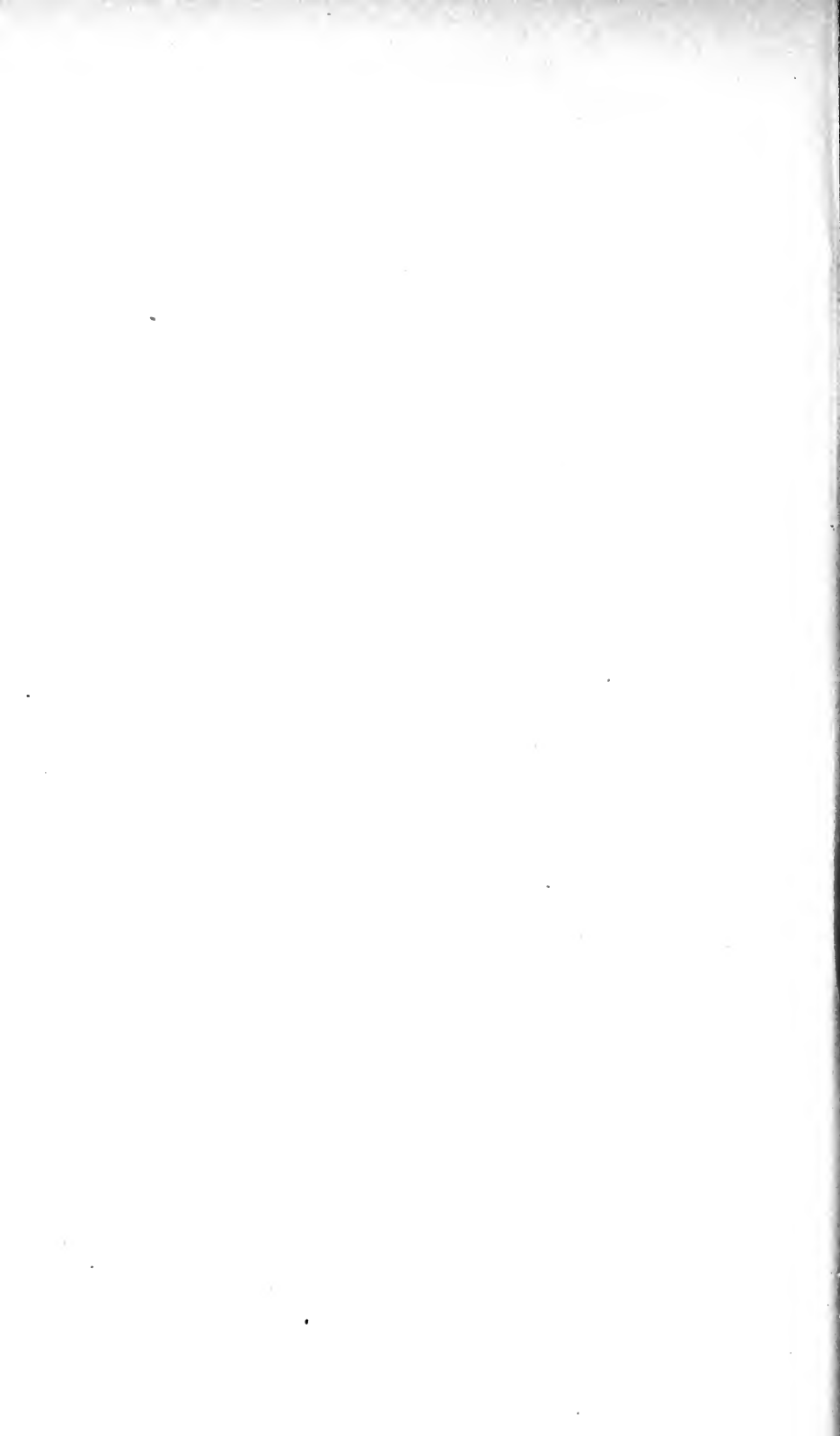
Name.	Symbol.	Atomic weight.	Name.	Symbol.	Atomic weight.
Aluminum.....	Al.....	27.1	Neodymium.....	Nd.....	143.6
Antimony.....	Sb.....	120.2	Neon.....	Ne.....	20.0
Argon.....	A.....	39.9	Nickel.....	Ni.....	58.7
Arsenic.....	As.....	75.0	Nitrogen.....	N.....	14.01
Barium.....	Ba.....	137.4	Osmium.....	Os.....	191.0
Bismuth.....	Bi.....	208.0	Oxygen.....	O.....	16.0
Boron.....	B.....	11.0	Palladium.....	Pd.....	106.5
Bromine.....	Br.....	79.96	Phosphorus.....	P.....	31.0
Cadmium.....	Cd.....	112.4	Platinum.....	Pt.....	194.8
Cæsium.....	Cs.....	132.9	Potassium.....	K.....	39.15
Calcium.....	Ca.....	40.1	Praseodymium.....	Pr.....	140.5
Carbon.....	C.....	12.00	Radium.....	Rd.....	225.0
Cerium.....	Ce.....	140.25	Rhodium.....	Rh.....	103.0
Chlorine.....	Cl.....	35.45	Rubidium.....	Rb.....	85.5
Chromium.....	Cr.....	52.1	Ruthenium.....	Ru.....	101.7
Cobalt.....	Co.....	59.0	Samarium.....	Sa.....	150.3
Columbium.....	Cb.....	94.0	Scandium.....	Sc.....	44.1
Copper.....	Cu.....	63.6	Selenium.....	Se.....	79.2
Erbium.....	Er.....	166.0	Silicon.....	Si.....	28.4
Europium.....	Eu.....	152.0	Silver.....	Ag.....	107.93
Fluorine.....	F.....	19.0	Sodium.....	Na.....	23.05
Gadolinium.....	Gd.....	156.0	Strontium.....	Sr.....	87.6
Gallium.....	Ga.....	70.0	Sulphur.....	S.....	32.06
Germanium.....	Ge.....	72.5	Tantalum.....	Ta.....	181.0
Glucinum.....	Gl.....	9.1	Tellurium.....	Te.....	127.6
Gold.....	Au.....	197.2	Terbium.....	Tb.....	159.2
Helium.....	He.....	4.0	Thallium.....	Tl.....	204.1
Hydrogen.....	H.....	1.008	Thorium.....	Th.....	232.5
Indium.....	In.....	115.0	Thulium.....	Tm.....	171.0
Iodine.....	I.....	126.97	Tin.....	Sn.....	119.0
Iridium.....	Ir.....	193.0	Titanium.....	Ti.....	48.1
Iron.....	Fe.....	55.9	Tungsten.....	W.....	184.0
Krypton.....	Kr.....	81.8	Uranium.....	U.....	238.5
Lanthanum.....	La.....	138.9	Vanadium.....	V.....	51.2
Lead.....	Pb.....	206.9	Xenon.....	Xe.....	128.0
Lithium.....	Li.....	7.03	Ytterbium.....	Yb.....	173.0
Magnesium.....	Mg.....	24.36	Yttrium.....	Yt.....	89.0
Manganese.....	Mn.....	55.0	Zinc.....	Zn.....	65.4
Mercury.....	Hg.....	200.0	Zirconium.....	Zr.....	90.6
Molybdenum.....	Mo.....	96.0			

<sup>a</sup> J. Amer. Chem. Soc., 1907, 29: 111.

TABLE XI.—*Conversion of nitrogen to protein.*

[N × 6.25 = Protein.]

N.	Protein.	N.	Protein.	N.	Protein.	N.	Protein.	N.	Protein.
<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>
0.01	0.06	0.71	4.44	1.41	8.81	2.11	13.19	2.81	17.56
.02	.13	.72	4.50	1.42	8.88	2.12	13.25	2.82	17.63
.03	.19	.73	4.56	1.43	8.94	2.13	13.31	2.83	17.69
.04	.25	.74	4.63	1.44	9.00	2.14	13.38	2.84	17.75
.05	.31	.75	4.69	1.45	9.06	2.15	13.44	2.85	17.81
.06	.38	.76	4.75	1.46	9.13	2.16	13.50	2.86	17.88
.07	.44	.77	4.81	1.47	9.19	2.17	13.56	2.87	17.94
.08	.50	.78	4.88	1.48	9.25	2.18	13.63	2.88	18.00
.09	.56	.79	4.94	1.49	9.31	2.19	13.69	2.89	18.06
.10	.63	.80	5.00	1.50	9.38	2.20	13.75	2.90	18.13
.11	.69	.81	5.06	1.51	9.44	2.21	13.81	2.91	18.19
.12	.75	.82	5.13	1.52	9.50	2.22	13.88	2.92	18.25
.13	.81	.83	5.19	1.53	9.56	2.23	13.94	2.93	18.31
.14	.88	.84	5.25	1.54	9.63	2.24	14.00	2.94	18.38
.15	.94	.85	5.31	1.55	9.69	2.25	14.06	2.95	18.44
.16	1.00	.86	5.38	1.56	9.75	2.26	14.13	2.96	18.50
.17	1.06	.87	5.44	1.57	9.81	2.27	14.19	2.97	18.56
.18	1.13	.88	5.50	1.58	9.88	2.28	14.25	2.98	18.63
.19	1.19	.89	5.56	1.59	9.94	2.29	14.31	2.99	18.69
.20	1.25	.90	5.63	1.60	10.00	2.30	14.38	3.00	18.75
.21	1.31	.91	5.69	1.61	10.06	2.31	14.44	3.01	18.81
.22	1.38	.92	5.75	1.62	10.13	2.32	14.50	3.02	18.88
.23	1.44	.93	5.81	1.63	10.19	2.33	14.56	3.03	18.94
.24	1.50	.94	5.88	1.64	10.25	2.34	14.63	3.04	19.00
.25	1.56	.95	5.94	1.65	10.31	2.35	14.69	3.05	19.06
.26	1.63	.96	6.00	1.66	10.38	2.36	14.75	3.06	19.13
.27	1.69	.97	6.06	1.67	10.44	2.37	14.81	3.07	19.19
.28	1.75	.98	6.13	1.68	10.50	2.38	14.88	3.08	19.25
.29	1.81	.99	6.19	1.69	10.56	2.39	14.94	3.09	19.31
.30	1.88	1.00	6.25	1.70	10.63	2.40	15.00	3.10	19.38
.31	1.94	1.01	6.31	1.71	10.69	2.41	15.06	3.11	19.44
.32	2.00	1.02	6.38	1.72	10.75	2.42	15.13	3.12	19.50
.33	2.06	1.03	6.44	1.73	10.81	2.43	15.19	3.13	19.56
.34	2.13	1.04	6.50	1.74	10.88	2.44	15.25	3.14	19.63
.35	2.19	1.05	6.56	1.75	10.94	2.45	15.31	3.15	19.69
.36	2.25	1.06	6.63	1.76	11.00	2.46	15.38	3.16	19.75
.37	2.31	1.07	6.69	1.77	11.06	2.47	15.44	3.17	19.81
.38	2.38	1.08	6.75	1.78	11.13	2.48	15.50	3.18	19.88
.39	2.44	1.09	6.81	1.79	11.19	2.49	15.56	3.19	19.94
.40	2.50	1.10	6.88	1.80	11.25	2.50	15.63	3.20	20.00
.41	2.56	1.11	6.94	1.81	11.31	2.51	15.69	3.21	20.06
.42	2.63	1.12	7.00	1.82	11.38	2.52	15.75	3.22	20.13
.43	2.69	1.13	7.06	1.83	11.44	2.53	15.81	3.23	20.19
.44	2.75	1.14	7.13	1.84	11.50	2.54	15.88	3.24	20.25
.45	2.81	1.15	7.19	1.85	11.56	2.55	15.94	3.25	20.31
.46	2.88	1.16	7.25	1.86	11.63	2.56	16.00	3.26	20.38
.47	2.94	1.17	7.31	1.87	11.69	2.57	16.06	3.27	20.44
.48	3.00	1.18	7.38	1.88	11.75	2.58	16.13	3.28	20.50
.49	3.06	1.19	7.44	1.89	11.81	2.59	16.19	3.29	20.56
.50	3.13	1.20	7.50	1.90	11.88	2.60	16.25	3.30	20.63
.51	3.19	1.21	7.56	1.91	11.94	2.61	16.31	3.31	20.69
.52	3.25	1.22	7.63	1.92	12.00	2.62	16.38	3.32	20.75
.53	3.31	1.23	7.69	1.93	12.06	2.63	16.44	3.33	20.81
.54	3.38	1.24	7.75	1.94	12.13	2.64	16.50	3.34	20.88
.55	3.44	1.25	7.81	1.95	12.19	2.65	16.56	3.35	20.94
.56	3.50	1.26	7.88	1.96	12.25	2.66	16.63	3.36	21.00
.57	3.56	1.27	7.94	1.97	12.31	2.67	16.69	3.37	21.06
.58	3.63	1.28	8.00	1.98	12.38	2.68	16.75	3.38	21.13
.59	3.69	1.29	8.06	1.99	12.44	2.69	16.81	3.39	21.19
.60	3.75	1.30	8.13	2.00	12.50	2.70	16.88	3.40	21.25
.61	3.81	1.31	8.19	2.01	12.56	2.71	16.94	3.41	21.31
.62	3.88	1.32	8.25	2.02	12.63	2.72	17.00	3.42	21.38
.63	3.94	1.33	8.31	2.03	12.69	2.73	17.06	3.43	21.44
.64	4.00	1.34	8.38	2.04	12.75	2.74	17.13	3.44	21.50
.65	4.06	1.35	8.44	2.05	12.81	2.75	17.19	3.45	21.56
.66	4.13	1.36	8.50	2.06	12.88	2.76	17.25	3.46	21.63
.67	4.19	1.37	8.56	2.07	12.94	2.77	17.31	3.47	21.69
.68	4.25	1.38	8.63	2.08	13.00	2.78	17.38	3.48	21.75
.69	4.31	1.39	8.69	2.09	13.06	2.79	17.44	3.49	21.81
.70	4.38	1.40	8.75	2.10	13.13	2.80	17.50	3.50	21.88





## APPENDIX.

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### **I. METHODS FOR THE ANALYSIS OF FERTILIZERS.**

#### **THOMAS OR BASIC SLAG.—PROVISIONAL.**

[Page 1.]

##### **1. Preparation of Sample.**

Prepare the sample for analysis as directed for other fertilizers or fertilizing materials (page 1, section 1).

##### **2. Phosphoric Acid.**

Make up the solution for analysis as directed in a<sub>7</sub>, page 2, under "(2) Total phosphoric acid," or in strong hydrochloric acid alone. In the latter case after the portion for analysis is measured out, add nitric acid and heat for a few minutes. Then determine total phosphoric acid according to the official methods (page 1, section 3).

##### **3. Fineness of Material.**

Determine the fineness of the material according to the plan followed with bone meal and estimate the commercial value on the basis of total phosphoric acid and fineness of product.

## II. METHODS FOR THE ANALYSIS OF SOILS.

### Total Organic Carbon.—Optional Official.

[Page 14, paragraph 4.]

Thoroughly mix 2 grams of soil (1 gram of soils high in organic matter), 0.75 gram of magnesium powder, and 10 grams of sodium peroxid, in a closed dry calorimeter bomb, by shaking the bomb back and forth. Explode the charge by means of electricity or by dropping a red-hot plug into the bomb through a valve which closes automatically as soon as the plug enters. Remove the fused charge from the bomb, using as small an amount of hot distilled water as possible, bring to a boil, and transfer to a receiving funnel of a Parr's apparatus for total carbon.<sup>a</sup>

From the acid funnel run 50 cc of sulphuric acid (1 part concentrated acid to 2 parts water) into a 150 cc Erlenmeyer flask. Connect the apparatus and slowly turn in the contents from the receiving funnel. The carbon dioxid generated passes through a capillary tube into a graduated burette.

Bring the contents of the flask to a boil, then fill the flask with distilled water from the receiving funnel so as to push the gases into the graduated burette. Note the temperature and pressure and the reading on the burette. Absorb the carbon dioxid by passing the gas into an ordinary absorption pipette which contains a 30 per cent potassium hydroxid solution. When a constant reading has been reached note the difference between it and the first reading, which gives the number of cubic centimeters of carbon dioxid equivalent to the carbon in sample.

Determine by the usual method the carbon dioxid in the soil as carbonates, using 10 grams of soil, and subtract the inorganic carbon from the total to find the organic carbon.

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### TOTAL PHOSPHORUS.

[Page 16, paragraph (f).]

#### (3) SODIUM PEROXID FUSION METHOD.—PROVISIONAL.

Weigh 10 grams of sodium peroxid into an iron or porcelain crucible and thoroughly mix with it 5 grams of the soil. If the soil is very low in organic matter, add a little starch to hasten the action. Heat the mixture carefully by applying the flame of a Bunsen burner directly upon the surface of the charge and the sides of the crucible until the action starts. Cover crucible until reaction is over and keep at a low red heat for fifteen minutes. Do not allow fusion to take place. By means of a large funnel and a stream of hot water, transfer the charge to a 500 cc measuring flask. Acidify with hydrochloric acid and boil. Let cool and make up to the mark. If the action has taken place properly there should be no particles of undecomposed soil in the bottom of the flask. Allow the silica to settle and draw off 200 cc of the clear solution.

Precipitate the iron, alumina, and phosphorus with ammonium hydroxid; filter, wash several times with hot water, return the precipitate to the beaker with a stream of hot water, holding the funnel over the beaker, and dissolve the precipitate in hot hydrochloric acid, pouring the acid upon the filter to dissolve any precipitate remain-

ing. Evaporate the solution and washings to complete dryness on a water bath. Take up with dilute hydrochloric acid, heating if necessary, and filter out the silica. Evaporate filtrate and washings to about 10 cc, add 2 cc of concentrated nitric acid, and just neutralize with ammonium hydroxid. Clear up with nitric acid, avoiding an excess. Heat at 40° to 50° on water bath, add 15 cc of molybdic solution, keeping at this temperature for from one to two hours. Let stand over night, filter, and wash free of acid with a one-tenth per cent solution of ammonium nitrate and, finally, once or twice with cold water. Transfer filter to beaker, and dissolve in standard potassium hydroxid (1 cc = 0.2 mg P), titrate the excess of potassium hydroxid with standard nitric acid, using phenolphthalein as indicator.

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### III. INORGANIC PLANT CONSTITUENTS.

#### Combustion Method.—Provisional.

[See page 21.]

Burn the material in a current of oxygen, using the form of combustion tube devised by Barlow.<sup>a</sup>

#### (1) APPARATUS.

Draw out at one end a combustion tube of Jena glass (60 or 70 cm in length and 1.5 cm in diameter) and bend it down. At about 30 cm from the bend fuse onto the combustion tube a side tube of the same kind of glass, having an internal diameter of 6 to 7 mm. Such a joint is made without special difficulty by a skilled glass-worker. The section of the main tube designated in fig. 1 as AD is from 30 to 40 cm, depending on the number of boats to be used and the amount of substance to be burnt. A convenient length for DB is 30 cm. This allows a little play at the end of the furnace, marked in the figure by the line HH. The lateral tube rises vertically about 3 or 4 cm so as to clear the furnace, and is then bent approximately at right angles so as to rest on the side rail of the furnace. Dress the tube in the following manner: At G place a perforated disk of platinum foil provided with a loop of platinum wire as a handle. Push into place by means of a long glass tube. A plug of asbestos may

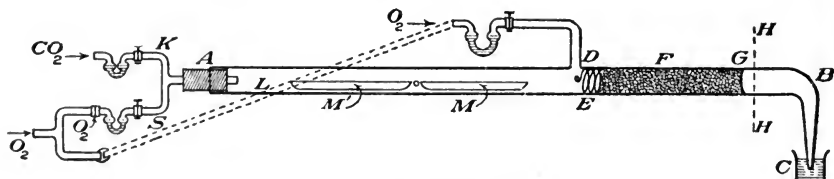


FIG. 1.—Combustion tube.

be substituted, but the platinum is cleaner and more convenient to handle in the subsequent operations. If asbestos is used, it must first be ignited in such a way that it does not come in contact with the naked Bunsen flames. Next fill in the column of absorbing material F, from 12 to 20 cm in length, depending on the amount and nature of the substance to be burned. For the absorbing material use sodium carbonate, free from sulphates. To avoid an excessive amount of salts in the final solution, use clean sand or bits of porcelain which have been impregnated with sodium carbonate and dried. In this manner an absorption column of 25 cm may be obtained without using more than 2 or 3 grams of the salt.

Press on top of the quartz-soda column, a spiral of platinum wire, E, one end of which is formed into a hook. It should fit the tube, so as to act as a light support for the column. The front of the spiral should be directly opposite the opening of the side-tube D. Introduce the weighed material, leaving a space about 5 or 6 cm long, between the front of the boats and the platinum spiral at E, and place the tube in the cold furnace. Insert the cork A, with the T-tube KS. Place a "safety-beaker" containing a little water at C, in order to furnish a test as to the completeness of the absorption. Connect the side-tube D with an oxygen supply, and the tubes K and

S with sources of carbon dioxid and oxygen, respectively. All of these gas-streams pass through small wash-bottles or bulbs to render the speed of the stream ascertainable, and are controlled by screw-clamps on short pieces of rubber tubing. Do not heat the part of the tube near the cork, and keep it free from any support in order to avoid the danger of charring the cork.

## (2) COMBUSTION.

Place from 1 to 5 grams in weighed platinum boats. Everything being arranged as described in section (1), close the clamps at S and D, controlling the oxygen. Pass a slow current of carbon dioxid in through K, and heat the part of the tube from E to H to a low red heat. It is advisable to have the temperature a little higher at E than at H, and diminish it gradually along the tube from E toward H, maintaining at the latter point a temperature below dull red heat. When the part EH is hot, admit a very slow stream of oxygen through D, the carbon dioxid stream being left unaltered. Next heat very gradually from E toward the boat M. Light one burner near A to prevent any back distillation. The instant the substance in the front part of the boat M begins to char, increase the oxygen stream through D, and after a short time—usually only a few seconds—the platinum spiral at E begins to glow, and the gases from M catch fire in the excess of oxygen and burn with a small flame at or near the spiral. After M is charred light burners under M'. At the beginning have a fairly strong oxygen stream going through

D. When the gases have caught fire, the adjustment of the stream at D is an easy matter, the position and shape of the disk of the flame itself indicating the state of affairs. In fig. 2 the line *ab* shows in section the position assumed by the disk of flame when the oxygen supply is properly adjusted. When the flame becomes vertical and travels off toward the substance, assuming the position *ef*, for example, a needless excess of oxygen is being used. On the other hand, if the oxygen be

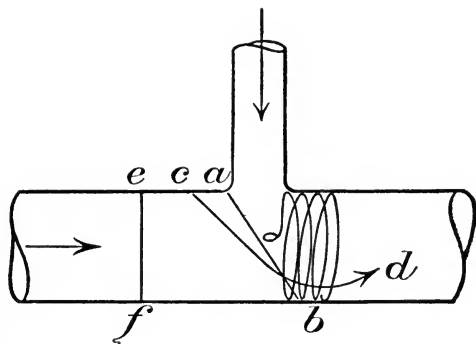


FIG. 2.—Detail of combustion tube.

insufficient, the gases from the char tilt the disk of flame into some such position as that represented by *cd*. In this case the lower edge *d* begins to flutter, the flame turns smoky, and carbon is deposited on the first portions of the sodium carbonate. The soot, however, almost instantly disappears if the oxygen supply is momentarily increased. A supply of oxygen which just prevents the deposition of soot is sufficient.

From this moment on, the combustion goes forward almost automatically. Gradually extend the heat toward the front end of the tube—the cork end—avoiding a high temperature. All that is necessary in this stage is the complete charring of the substance, and this takes place below red heat. The gases from the charring mass are carried by the stream of carbon dioxid (which may now be made very slow) to the flame. It will be seen at once that it is impossible for an explosive mixture to be formed between D and A, since the gases are prevented by the carbon dioxid stream from mixing with oxygen until they reach the surface of the flame, beyond which sharply defined surface there is a large excess of oxygen.

When the whole substance is charred, the flame assumes a vertical position and travels slowly toward the boat. If an attempt is now made to bring it back to *ab* by diminishing the supply of oxygen, the flame grows smaller, flickers, and goes out. At this point turn up the burners along the section from D to A, shut off the carbon dioxid,

and pass a stream of oxygen through S, the oxygen stream at D being diminished, but not entirely shut off. When the char has gained a proper temperature, the end nearest A takes fire in the oxygen and the mass glows steadily from A toward D. When the carbon is all consumed, stop the oxygen supply at D, diminish that at S, and gradually lower the temperature during ten minutes, then shut off the burners. Stop the oxygen, and allow the tube to cool somewhat, passing in a slow stream of carbon dioxid. The charring should always proceed slowly, using an excess of oxygen. The time required for one combustion is about one hour. When the tube is sufficiently cool, remove boats, cool in desiccator, and weigh. The total weight less the weight of the boats equals the ash. For complete analysis of ash proceed as under "III. Inorganic Plant Constituents," page 21, (c), (d), et seq.

Remove the sodium carbonate containing the volatile products from the tube. Extract with hot water and hydrochloric acid, remove silica, and determine sulphuric and phosphoric acids and chlorin as directed under "III. Inorganic Plant Constituents," pages 22 and 23, paragraphs 4, 5, and 6.

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PREPARATION OF ASH WITHOUT THE USE OF CALCIUM ACETATE.—OPTIONAL OFFICIAL

[Page 21 (b)].

Before combustion thoroughly clean the material from all foreign matter. Conduct the combustion at a comparatively low temperature, never employing a full red heat (because of the danger of volatilizing alkaline chlorids, etc.) nor a strong draft of air, lest the lighter part of the ash be carried away. Preferably employ a flat platinum dish in a muffle. With substances rich in silica and alkalis it is better first to char the substances, wash with distilled water to remove soluble salts, then dry and incinerate the residue. Evaporate the aqueous extract and add it to the ash. With substances rich in phosphates, e. g., seeds and animal substances, char the material, remove salts by acetic acid, decant the acetic solution, wash with distilled water, and then complete the combustion. Add the acetic solution and washings to the ash, evaporate to dryness, and gently ignite the whole to decompose the acetates. In whatever way obtained, the whole of the ash should be pulverized and intimately mixed while still warm, and preserved in a tight, dry bottle for analysis. If after incineration the ash has absorbed moisture, dry thoroughly at low redness before bottling.

## IV. METHODS FOR THE ANALYSIS OF INSECTICIDES AND FUNGICIDES.

### 11. Lead Arsenate (Haywood).—Provisional.

[Page 34.]

#### (a) PREPARATION OF SAMPLE.

In case the sample is in the form of a paste, as it usually is, dry the whole of it to constant weight at the temperature of boiling water and calculate the result as total moisture. Grind the dry sample (which will gain a small amount of moisture by so doing) to a fine powder and determine the various constituents as follows:

#### (b) MOISTURE.

Weigh 2 grams of the sample and heat in the water bath for eight hours or in the hot-air bath at 110° C. for five to six hours or till constant weight is obtained.

#### (c) TOTAL LEAD OXID.

Dissolve 2 grams of the sample in about 80 cc of water and 15 cc of concentrated nitric acid on the steam bath; transfer the solution to a 250 cc flask and make up to the mark. To 50 cc of the solution add 3 cc of concentrated sulphuric acid, evaporate on the steam bath to a sirupy consistency and then on the hot plate till white fumes appear and all nitric acid has been given off. Add 50 cc of water and 100 cc of 95 per cent alcohol. Let stand for several hours and filter off supernatant liquid, wash about ten times with acidified alcohol (water 100 parts, 95 per cent alcohol 200 parts, and concentrated sulphuric acid 3 parts) and then with 95 per cent alcohol till free of sulphuric acid. Dry, remove as much as possible of the precipitate from the paper into a weighed crucible, and ignite at low red heat. Burn the paper in a separate porcelain crucible and treat the residue first with a little nitric acid, which is afterwards evaporated off, and then with a drop or two of sulphuric acid. Ignite, weigh, and add this weight to the weight of the precipitate previously removed from the paper for amount of the lead sulphate.

#### (d) TOTAL ARSENIC OXID (MODIFIED GOOCH AND BROWNING METHOD *a*).

Transfer 100 cc of the nitric acid solution of the sample, prepared as in the above determination of lead, to a porcelain dish, add 6 cc of concentrated sulphuric acid, evaporate to a sirupy consistency on water bath and then on hot plate to the appearance of white fumes of sulphuric acid. Wash into a 100 cc flask with water, make up to mark, filter through dry filters, and use 50 cc aliquot for further work. Transfer this to an Erlenmeyer flask of 400 cc capacity, add 4 cc of concentrated sulphuric acid and 1 gram of potassium iodid, dilute to about 100 cc and boil until the volume is reduced to about 40 cc. Cool the solution under running water, dilute to about 300 cc, and exactly use up the iodine set free and still remaining in solution with a few drops of approximately tenth-normal sodium thiosulphate. Wash the mixture into a large beaker, make alkaline with sodium carbonate, and slightly acidify with dilute sulphuric acid; then make alkaline again with an excess of sodium bicarbonate. Titrate the solution with a twentieth-normal iodine solution to the appearance of a blue color, using starch as indicator.

<sup>a</sup> Amer. J. Sci., 1890, 40: 66.

## (e) WATER-SOLUBLE LEAD OXID.

Place 2 grams of the lead arsenate in a flask with 2,000 cc of carbon dioxid free water and let stand ten days, shaking eight times a day. Filter through a dry filter and use aliquots of this for determining soluble lead and arsenic oxids and soluble solids; determine lead as described above for total lead, using the same relative proportions of sulphuric acid, water, and alcohol, but keeping the volume as small as possible.

## (f) WATER-SOLUBLE ARSENIC OXID.

For this determination use 200 to 400 cc of the water extract obtained under the determination of soluble lead oxid. Add 0.5 cc of sulphuric acid and evaporate it to a sirupy consistency, then heat on hot plate to appearance of white fumes. Add a very small amount of water and filter off lead through the very smallest filter paper, using as little wash water as possible. Place this filtrate in an Erlenmeyer flask, and determine arsenic as described under total arsenic oxid, using the same amount of reagents and the same dilutions.

## (g) SOLUBLE SOLIDS OR IMPURITIES.

Evaporate 200 cc of the water extract obtained above to dryness in a weighed platinum dish, dry to constant weight at the temperature of the boiling water bath, and weigh. The soluble solids so obtained represent principally any sodium acetate or sodium nitrate present, with a very small quantity, perhaps, of lead acetate or nitrate and some soluble arsenic, probably in the form of lead arsenate.

## (h) FORM OF STATING RESULTS.

	Per cent.
Moisture.....	.....
Total arsenic oxid.....	.....
Total lead oxid.....	.....
Soluble impurities (exclusive of soluble lead oxid and arsenic oxid).....	.....
Total.....	.....
Soluble arsenic oxid.....	.....
Soluble lead oxid.....	.....



## VI. GENERAL METHODS FOR THE ANALYSIS OF FOODS AND FEEDING STUFFS.

### Estimation of Reduced Copper.

[Page 51.]

LOW'S VOLUMETRIC METHOD, MODIFIED.<sup>a</sup>—PROVISIONAL.

#### (a) STANDARDIZATION OF THE THIOSULPHATE SOLUTION.

Prepare a solution of sodium thiosulphate containing 19 grams of pure crystals to 1,000 cc. Weigh accurately about 0.2 gram of pure copper foil and place in a flask of 250 cc capacity. Dissolve by warming with 5 cc of a mixture of equal volumes of strong nitric acid and water. Dilute to 50 cc, boil to expel the red fumes, add 5 cc strong bromin water, and boil until the bromin is thoroughly expelled. Remove from the heat and add a slight excess of strong ammonium hydroxid—7 cc is about the right amount. Again boil until the excess of ammonia is expelled, as shown by a change of color of the liquid, and a partial precipitation. Now add a slight excess of strong acetic acid (3 or 4 cc of 80 per cent acid) and boil for a minute. Cool to room temperature and add 10 cc of a solution of pure potassium iodid containing 300 grams of potassium iodid to 1,000 cc. Titrate at once with the thiosulphate solution until the brown tinge has become weak, then add sufficient starch liquor to produce a marked blue coloration. Continue the titration cautiously until the color due to free iodin has entirely vanished. The blue color changes toward the end to a faint lilac. If at this point the thiosulphate be added drop by drop and a little time be allowed for complete reaction after each addition there is no difficulty in determining the end point within a single drop. One cubic centimeter of the thiosulphate solution will be found to correspond to about 0.005 gram of copper.

#### (b) DETERMINATION OF COPPER.

After washing the precipitated cuprous oxid, cover the gooch with a watch glass and dissolve the oxid by means of 5 cc of warm nitric acid (1:1) poured under the watch glass with a pipette. Catch the filtrate in a flask of 250 cc capacity, wash watch glass and gooch free of copper; 50 cc of water will be sufficient. Boil to expel red fumes, add 5 cc of bromin water, boil off the bromin, and proceed exactly as in standardizing the thiosulphate.

#### (d) UNIFORM METHOD FOR DETERMINING REDUCING SUGARS IN GENERAL (MUNSON AND WALKER).<sup>b</sup>—PROVISIONAL.

[Page 42.]

#### (1) PREPARATION OF SOLUTIONS AND ASBESTOS.

(a) *Solutions*.—Use solutions (a), (b), and (c) as given on page 42, under Soxhlet's modification of Fehling's solution.

(b) *Asbestos*.—Prepare the asbestos, which should be the amphibole variety, by first digesting with 1:3 hydrochloric acid for two or three days. Wash free from acid

<sup>a</sup> J. Amer. Chem. Soc., 1902, 24: 1082.

<sup>b</sup> Ibid., 1906, 28: 663; 1907, 29: 541.

and digest for a similar period with soda solution, after which treat for a few hours with hot alkaline copper tartrate solution of the strength employed in sugar determinations. Then wash the asbestos free from alkali, finally digest with nitric acid for several hours, and after washing free from acid shake with water for use. In preparing the gooch crucible load it with a film of asbestos one-fourth inch thick, wash this thoroughly with water to remove fine particles of asbestos; finally wash with alcohol and ether, dry for thirty minutes at  $100^{\circ}\text{C}$ ., cool in a desiccator and weigh. It is best to dissolve the cuprous oxid with nitric acid each time after weighing and use the same felts over and over again, as they improve with use.

(2) DETERMINATION.

Transfer 25 cc each of the copper and alkaline tartrate solutions to a 400 cc Jena or Non-sol beaker and add 50 cc of reducing sugar solution, or, if a smaller volume of sugar solution be used, add water to make the final volume 100 cc. Heat the beaker upon an asbestos gauze over a Bunsen burner, so regulate the flame that boiling begins in four minutes, and continue the boiling for exactly two minutes. Keep the beaker covered with a watch-glass throughout the entire time of heating. Without diluting, filter the cuprous oxid at once on an asbestos felt in a porcelain gooch crucible, using suction. Wash the cuprous oxid thoroughly with water at a temperature of about  $60^{\circ}\text{C}$ ., then with 10 cc of alcohol and finally with 10 cc of ether. Dry for thirty minutes in a water oven at  $100^{\circ}\text{C}$ ., cool in a desiccator and weigh as cuprous oxid.

N. B. The number of milligrams of copper reduced by a given amount of reducing sugar differs when sucrose is present and when it is absent. In the tables following the absence of sucrose is assumed except in the two columns under invert sugar, where one for mixtures of invert sugar and sucrose (0.4 gram of total sugar in 50 cc of solution) and one for invert sugar and sucrose when the 50 cc of solution contains 2 grams of total sugar are given, in addition to the column for invert sugar alone.

Table for calculating dextrose, invert sugar alone, invert sugar in the presence of sucrose (0.4 gram and 2 grams total sugar), lactose (three forms), and maltose (anhydrous and crystallized).

[Expressed in milligrams.]

Cuprous oxid ( $\text{Cu}_2\text{O}$ ).	Copper (Cu).	Dextrose (d-glucose).	Invert sugar.	Invert sugar and sucrose.		Lactose.			Maltose.		Cuprous oxid ( $\text{Cu}_2\text{O}$ ).
				0.4 gram total sugar.	2 grams total sugar.	$\text{C}_{12}\text{H}_{22}\text{O}_{11}$ .	$\text{C}_{12}\text{H}_{22}\text{O}_{11} + \frac{1}{2} \text{H}_2\text{O}$ .	$\text{C}_{12}\text{H}_{22}\text{O}_{11} + \text{H}_2\text{O}$ .	$\text{C}_{12}\text{H}_{22}\text{O}_{11}$ .	$\text{C}_{12}\text{H}_{22}\text{O}_{11} + \text{H}_2\text{O}$ .	
10	8.9	4.0	4.5	1.6	.....	3.8	3.9	4.0	5.9	6.2	10
11	9.8	4.5	5.0	2.1	.....	4.5	4.6	4.7	6.7	7.0	11
12	10.7	4.9	5.4	2.5	.....	5.1	5.3	5.4	7.5	7.9	12
13	11.5	5.3	5.8	3.0	.....	5.8	5.9	6.1	8.3	8.7	13
14	12.4	5.7	6.3	3.4	.....	6.4	6.6	6.8	9.1	9.5	14
15	13.3	6.2	6.7	3.9	.....	7.1	7.3	7.5	9.9	10.4	15
16	14.2	6.6	7.2	4.3	.....	7.7	8.0	8.2	10.6	11.2	16
17	15.1	7.0	7.6	4.8	.....	8.4	8.6	8.8	11.4	12.0	17
18	16.0	7.5	8.1	5.2	.....	9.1	9.3	9.5	12.2	12.9	18
19	16.9	7.9	8.5	5.7	.....	9.7	10.0	10.2	13.0	13.7	19
20	17.8	8.3	8.9	6.1	.....	10.4	10.6	10.9	13.8	14.6	20
21	18.7	8.7	9.4	6.6	.....	11.0	11.3	11.6	14.6	15.4	21
22	19.5	9.2	9.8	7.0	.....	11.7	12.0	12.3	15.4	16.2	22
23	20.4	9.6	10.3	7.5	.....	12.3	12.7	13.0	16.2	17.1	23
24	21.3	10.0	10.7	7.9	.....	13.0	13.3	13.7	17.0	17.9	24
25	22.2	10.5	11.2	8.4	.....	13.6	14.0	14.4	17.8	18.7	25
26	23.1	10.9	11.6	8.8	.....	14.3	14.7	15.1	18.6	19.6	26
27	24.0	11.3	12.0	9.3	.....	15.0	15.3	15.7	19.4	20.4	27
28	24.9	11.8	12.5	9.7	.....	15.6	16.0	16.4	20.2	21.2	28
29	25.8	12.2	12.9	10.2	.....	16.3	16.7	17.1	21.0	22.1	29
30	26.6	12.6	13.4	10.7	4.3	16.9	17.4	17.8	21.8	22.9	30
31	27.5	13.1	13.8	11.1	4.7	17.6	18.0	18.5	22.6	23.7	31
32	28.4	13.5	14.3	11.6	5.2	18.2	18.7	19.2	23.3	24.6	32
33	29.3	13.9	14.7	12.0	5.6	18.9	19.4	19.9	24.1	25.4	33
34	30.2	14.3	15.2	12.5	6.1	19.5	20.1	20.6	24.9	26.2	34
35	31.1	14.8	15.6	12.9	6.5	20.2	20.7	21.3	25.7	27.1	35
36	32.0	15.2	16.1	13.4	7.0	20.9	21.4	22.0	26.5	27.9	36
37	32.9	15.6	16.5	13.8	7.4	21.5	22.1	22.7	27.3	28.7	37
38	33.8	16.1	16.9	14.3	7.9	22.2	22.8	23.3	28.1	29.6	38
39	34.6	16.5	17.4	14.7	8.4	22.8	23.4	24.0	28.9	30.4	39
40	35.5	16.9	17.8	15.2	8.8	23.5	24.1	24.7	29.7	31.3	40
41	36.4	17.4	18.3	15.6	9.3	24.1	24.8	25.4	30.5	32.1	41
42	37.3	17.8	18.7	16.1	9.7	24.8	25.4	26.1	31.3	32.9	42
43	38.2	18.2	19.2	16.6	10.2	25.4	26.1	26.8	32.1	33.8	43
44	39.1	18.7	19.6	17.0	10.7	26.1	26.8	27.5	32.9	34.6	44
45	40.0	19.1	20.1	17.5	11.1	26.8	27.5	28.2	33.7	35.4	45
46	40.9	19.6	20.5	17.9	11.6	27.4	28.1	28.8	34.4	36.3	46
47	41.7	20.0	21.0	18.4	12.0	28.1	28.8	29.5	35.2	37.1	47
48	42.6	20.4	21.4	18.8	12.5	28.7	29.5	30.2	36.0	37.9	48
49	43.5	20.9	21.9	19.3	12.9	29.4	30.1	30.9	36.8	38.8	49
50	44.4	21.3	22.3	19.7	13.4	30.0	30.8	31.6	37.6	39.6	50
51	45.3	21.7	22.8	20.2	13.9	30.7	31.5	32.3	38.4	40.4	51
52	46.2	22.2	23.2	20.7	14.3	31.3	32.1	33.0	39.2	41.3	52
53	47.1	22.6	23.7	21.1	14.8	32.0	32.8	33.6	40.0	42.1	53
54	48.0	23.0	24.1	21.6	15.2	32.6	33.5	34.3	40.8	42.9	54
55	48.9	23.5	24.6	22.0	15.7	33.3	34.1	35.0	41.6	43.8	55
56	49.7	23.9	25.0	22.5	16.2	33.9	34.8	35.7	42.4	44.6	56
57	50.6	24.3	25.5	22.9	16.6	34.6	35.5	36.4	43.2	45.4	57
58	51.5	24.8	25.9	23.4	17.1	35.2	36.1	37.1	44.0	46.3	58
59	52.4	25.2	26.4	23.9	17.5	35.9	36.8	37.7	44.8	47.1	59
60	53.3	25.6	26.8	24.3	18.0	36.5	37.5	38.4	45.6	48.0	60
61	54.2	26.1	27.3	24.8	18.5	37.2	38.2	39.1	46.3	48.8	61
62	55.1	26.5	27.7	25.2	18.9	37.8	38.8	39.8	47.1	49.6	62
63	56.0	27.0	28.2	25.7	19.4	38.5	39.5	40.5	47.9	50.5	63
64	56.8	27.4	28.6	26.2	19.8	39.2	40.2	41.2	48.7	51.3	64

Table for calculating dextrose, invert sugar alone, invert sugar in the presence of sucrose (0.4 gram and 2 grams total sugar), lactose (three forms), and maltose (anhydrous and crystallized)—Continued.

[Expressed in milligrams.]

Cuprous oxid ( $\text{Cu}_2\text{O}$ ).	Copper (Cu).	Dextrose ( <i>d</i> -glucose).	Invert sugar.	Invert sugar and sucrose.		Lactose.			Maltose.		Cuprous oxid ( $\text{Cu}_2\text{O}$ ).
				0.4 gram total sugar.	2 grams total sugar.	$\text{C}_{12}\text{H}_{22}\text{O}_{11}$ .	$\text{C}_{12}\text{H}_{22}\text{O}_{11} + \frac{1}{3} \text{H}_2\text{O}$ .	$\text{C}_{12}\text{H}_{22}\text{O}_{11} + \text{H}_2\text{O}$ .	$\text{C}_{12}\text{H}_{22}\text{O}_{11}$ .	$\text{C}_{12}\text{H}_{22}\text{O}_{11} + \text{H}_2\text{O}$ .	
65	57.7	27.8	29.1	26.6	20.3	39.8	40.9	41.9	49.5	52.1	65
66	58.6	28.3	29.5	27.1	20.8	40.5	41.6	42.6	50.3	53.0	66
67	59.5	28.7	30.0	27.5	21.2	41.1	42.2	43.3	51.1	53.8	67
68	60.4	29.2	30.4	28.0	21.7	41.8	42.9	44.0	51.9	54.6	68
69	61.3	29.6	30.9	28.5	22.2	42.5	43.6	44.7	52.7	55.5	69
70	62.2	30.0	31.3	28.9	22.6	43.1	44.3	45.4	53.5	56.3	70
71	63.1	30.5	31.8	29.4	23.1	43.8	44.9	46.1	54.3	57.1	71
72	64.0	30.9	32.3	29.8	23.5	44.4	45.6	46.8	55.1	58.0	72
73	64.8	31.4	32.7	30.3	24.0	45.1	46.3	47.5	55.9	58.8	73
74	65.7	31.8	33.2	30.8	24.5	45.7	47.0	48.2	56.7	59.6	74
75	66.6	32.2	33.6	31.2	24.9	46.4	47.6	48.8	57.5	60.5	75
76	67.5	32.7	34.1	31.7	25.4	47.0	48.3	49.5	58.2	61.3	76
77	68.4	33.1	34.5	32.1	25.9	47.7	49.0	50.2	59.0	62.1	77
78	69.3	33.6	35.0	32.6	26.3	48.4	49.6	50.9	59.8	63.0	78
79	70.2	34.0	35.4	33.1	26.8	49.0	50.3	51.6	60.6	63.8	79
80	71.1	34.4	35.9	33.5	27.3	49.7	51.0	52.3	61.4	64.6	80
81	71.9	34.9	36.3	34.0	27.7	50.3	51.6	53.0	62.2	65.5	81
82	72.8	35.3	36.8	34.5	28.2	51.0	52.3	53.7	63.0	66.3	82
83	73.7	35.8	37.3	34.9	28.6	51.6	53.0	54.4	63.8	67.1	83
84	74.6	36.2	37.7	35.4	29.1	52.3	53.7	55.0	64.6	68.0	84
85	75.5	36.7	38.2	35.8	29.6	52.9	54.3	55.7	65.4	68.8	85
86	76.4	37.1	38.6	36.3	30.0	53.6	55.0	56.4	66.2	69.7	86
87	77.3	37.5	39.1	36.8	30.5	54.3	55.7	57.1	67.0	70.5	87
88	78.2	38.0	39.5	37.2	31.0	54.9	56.4	57.8	67.8	71.3	88
89	79.1	38.4	40.0	37.7	31.4	55.6	57.0	58.5	68.5	72.2	89
90	79.9	38.9	40.4	38.2	31.9	56.2	57.7	59.2	69.3	73.0	90
91	80.8	39.3	40.9	38.6	32.4	56.9	58.4	59.9	70.1	73.8	91
92	81.7	39.8	41.4	39.1	32.8	57.5	59.0	60.6	70.9	74.7	92
93	82.6	40.2	41.8	39.6	33.3	58.2	59.7	61.3	71.7	75.5	93
94	83.5	40.6	42.3	40.0	33.8	58.8	60.4	61.9	72.5	76.3	94
95	84.4	41.1	42.7	40.5	34.2	59.5	61.1	62.6	73.3	77.2	95
96	85.3	41.5	43.2	41.0	34.7	60.2	61.7	63.3	74.1	78.0	96
97	86.2	42.0	43.7	41.4	35.2	60.8	62.4	64.0	74.9	78.8	97
98	87.1	42.4	44.1	41.9	35.6	61.5	63.1	64.7	75.7	79.7	98
99	87.9	42.9	44.6	42.4	36.1	62.1	63.8	65.4	76.5	80.5	99
100	88.8	43.3	45.0	42.8	36.6	62.8	64.4	66.1	77.3	81.3	100
101	89.7	43.8	45.5	43.3	37.0	63.4	65.1	66.8	78.1	82.2	101
102	90.6	44.2	46.0	43.8	37.5	64.1	65.8	67.5	78.8	83.0	102
103	91.5	44.7	46.4	44.2	38.0	64.7	66.4	68.1	79.6	83.8	103
104	92.4	45.1	46.9	44.7	38.5	65.4	67.1	68.8	80.4	84.7	104
105	93.3	45.5	47.3	45.2	38.9	66.1	67.8	69.5	81.2	85.5	105
106	94.2	46.0	47.8	45.6	39.4	66.7	68.5	70.2	82.0	86.3	106
107	95.0	46.4	48.3	46.1	39.9	67.4	69.1	70.9	82.8	87.2	107
108	95.9	46.9	48.7	46.6	40.3	68.0	69.8	71.6	83.6	88.0	108
109	96.8	47.3	49.2	47.0	40.8	68.7	70.5	72.3	84.4	88.8	109
110	97.7	47.8	49.6	47.5	41.3	69.3	71.1	73.0	85.2	89.7	110
111	98.6	48.2	50.1	48.0	41.7	70.0	71.8	73.6	86.0	90.5	111
112	99.5	48.7	50.6	48.4	42.2	70.6	72.5	74.3	86.8	91.3	112
113	100.4	49.1	51.0	48.9	42.7	71.3	73.1	75.0	87.6	92.2	113
114	101.3	49.6	51.5	49.4	43.2	71.9	73.8	75.7	88.4	93.0	114
115	102.2	50.0	51.9	49.8	43.6	72.6	74.5	76.4	89.2	93.9	115
116	103.0	50.5	52.4	50.3	44.1	73.2	75.2	77.1	90.0	94.7	116
117	103.9	50.9	52.9	50.8	44.6	73.9	75.8	77.8	90.7	95.5	117
118	104.8	51.4	53.3	51.2	45.0	74.5	76.5	78.5	91.5	96.4	118
119	105.7	51.8	53.8	51.7	45.5	75.2	77.2	79.1	92.3	97.2	119

Table for calculating dextrose, invert sugar alone, invert sugar in the presence of sucrose (0.4 gram and 2 grams total sugar), lactose (three forms), and maltose (anhydrous and crystallized)—Continued.

[Expressed in milligrams.]

Cuprous oxid ( $\text{Cu}_2\text{O}$ ).	Copper (Cu).	Dextrose ( $d$ -glucose).	Invert sugar.	Invert sugar • and sucrose.		Lactose.			Maltose.		Cuprous oxid ( $\text{Cu}_2\text{O}$ ).
				0.4 gram total sugar.	2 grams total sugar.	$\text{C}_{12}\text{H}_{22}\text{O}_{11}$ .	$\text{C}_{12}\text{H}_{22}\text{O}_{11} + \frac{1}{2} \text{H}_2\text{O}$ .	$\text{C}_{12}\text{H}_{22}\text{O}_{11} + \text{H}_2\text{O}$ .	$\text{C}_{12}\text{H}_{22}\text{O}_{11}$ .	$\text{C}_{12}\text{H}_{22}\text{O}_{11} + \text{H}_2\text{O}$ .	
120	106.6	52.3	54.3	52.2	46.0	75.8	77.8	79.8	93.1	98.0	120
121	107.5	52.7	54.7	52.7	46.5	76.5	78.5	80.5	93.9	98.9	121
122	108.4	53.2	55.2	53.1	46.9	77.1	79.2	81.2	94.7	99.7	122
123	109.3	53.6	55.7	53.6	47.4	77.8	79.9	81.9	95.5	100.5	123
124	110.1	54.1	56.1	54.1	47.9	78.5	80.5	82.6	96.3	101.4	124
125	111.0	54.5	56.6	54.5	48.3	79.1	81.2	83.3	97.1	102.2	125
126	111.9	55.0	57.0	55.0	48.8	79.8	81.9	84.0	97.9	103.0	126
127	112.8	55.4	57.5	55.5	49.3	80.4	82.5	84.7	98.7	103.9	127
128	113.7	55.9	58.0	55.9	49.8	81.1	83.2	85.4	99.4	104.7	128
129	114.6	56.3	58.4	56.4	50.2	81.7	83.9	86.0	100.2	105.5	129
130	115.5	56.8	58.9	56.9	50.7	82.4	84.6	86.7	101.0	106.4	130
131	116.4	57.2	59.4	57.4	51.2	83.1	85.2	87.4	101.8	107.2	131
132	117.3	57.7	59.8	57.8	51.7	83.7	85.9	88.1	102.6	108.0	132
133	118.1	58.1	60.3	58.3	52.1	84.4	86.6	88.8	103.4	108.9	133
134	119.0	58.6	60.8	58.8	52.6	85.0	87.3	89.5	104.2	109.7	134
135	119.9	59.0	61.2	59.3	53.1	85.7	87.9	90.2	105.0	110.5	135
136	120.8	59.5	61.7	59.7	53.6	86.3	88.6	90.9	105.8	111.4	136
137	121.7	60.0	62.2	60.2	54.0	87.0	89.3	91.6	106.6	112.2	137
138	122.6	60.4	62.6	60.7	54.5	87.7	90.0	92.3	107.4	113.0	138
139	123.5	60.9	63.1	61.2	55.0	88.3	90.6	93.0	108.2	113.9	139
140	124.4	61.3	63.6	61.6	55.5	89.0	91.3	93.6	109.0	114.7	140
141	125.2	61.8	64.0	62.1	55.9	89.6	92.0	94.3	109.8	115.5	141
142	126.1	62.2	64.5	62.6	56.4	90.3	92.6	95.0	110.5	116.4	142
143	127.0	62.7	65.0	63.1	56.9	90.9	93.3	95.7	111.3	117.2	143
144	127.9	63.1	65.4	63.5	57.4	91.6	94.0	96.4	112.1	118.0	144
145	128.8	63.6	65.9	64.0	57.8	92.2	94.7	97.1	112.9	118.9	145
146	129.7	64.0	66.4	64.5	58.3	92.9	95.3	97.8	113.7	119.7	146
147	130.6	64.5	66.9	65.0	58.8	93.5	96.0	98.4	114.5	120.5	147
148	131.5	65.0	67.3	65.4	59.3	94.2	96.7	99.1	115.3	121.4	148
149	132.4	65.4	67.8	65.9	59.7	94.8	97.3	99.8	116.1	122.2	149
150	133.2	65.9	68.3	66.4	60.2	95.5	98.0	100.5	116.9	123.0	150
151	134.1	66.3	68.7	66.9	60.7	96.2	98.7	101.2	117.7	123.9	151
152	135.0	66.8	69.2	67.3	61.2	96.8	99.3	101.9	118.5	124.7	152
153	135.9	67.2	69.7	67.8	61.7	97.5	100.0	102.6	119.3	125.5	153
154	136.8	67.7	70.1	68.3	62.1	98.1	100.7	103.3	120.0	126.4	154
155	137.7	68.2	70.6	68.8	62.6	98.8	101.4	104.0	120.8	127.2	155
156	138.6	68.6	71.1	69.2	63.1	99.4	102.0	104.7	121.6	128.0	156
157	139.5	69.1	71.6	69.7	63.6	100.1	102.7	105.3	122.4	128.9	157
158	140.3	69.5	72.0	70.2	64.1	100.7	103.4	106.0	123.2	129.7	158
159	141.2	70.0	72.5	70.7	64.5	101.4	104.1	106.7	124.0	130.5	159
160	142.1	70.4	73.0	71.2	65.0	102.0	104.7	107.4	124.8	131.4	160
161	143.0	70.9	73.4	71.6	65.5	102.7	105.4	108.1	125.6	132.2	161
162	143.9	71.4	73.9	72.1	66.0	103.4	106.1	108.8	126.4	133.0	162
163	144.8	71.8	74.4	72.6	66.5	104.0	106.7	109.5	127.2	133.9	163
164	145.7	72.3	74.9	73.1	66.9	104.7	107.4	110.2	128.0	134.7	164
165	146.6	72.8	75.3	73.6	67.4	105.3	108.1	110.9	128.8	135.5	165
166	147.5	73.2	75.8	74.0	67.9	106.0	108.8	111.5	129.6	136.4	166
167	148.3	73.7	76.3	74.5	68.4	106.6	109.4	112.2	130.3	137.2	167
168	149.2	74.1	76.8	75.0	68.9	107.3	110.1	112.9	131.1	138.0	168
169	150.1	74.6	77.2	75.5	69.3	107.9	110.8	113.6	131.9	138.9	169
170	151.0	75.1	77.7	76.0	69.8	108.6	111.4	114.3	132.7	139.7	170
171	151.9	75.5	78.2	76.4	70.3	109.2	112.1	115.0	133.5	140.5	171
172	152.8	76.0	78.7	76.9	70.8	109.9	112.8	115.7	134.3	141.4	172
173	153.7	76.4	79.1	77.4	71.3	110.5	113.5	116.4	135.1	142.2	173
174	154.6	76.9	79.6	77.9	71.7	111.2	114.1	117.1	135.9	143.0	174

Table for calculating dextrose, invert sugar alone, invert sugar in the presence of sucrose (0.4 gram and 2 grams total sugar), lactose (three forms), and maltose (anhydrous and crystallized)—Continued.

[Expressed in milligrams.]

Cuprous oxid ( $\text{Cu}_2\text{O}$ ).	Copper (Cu).	Dextrose ( <i>d</i> -glucose).	Invert sugar.	Invert sugar and sucrose.		Lactose.			Maltose.		Cuprous oxid ( $\text{Cu}_2\text{O}$ ).
				0.4 gram total sugar.	2 grams total sugar.	$\text{C}_{12}\text{H}_{22}\text{O}_{11}$ .	$\text{C}_{12}\text{H}_{22}\text{O}_{11} + \frac{1}{2} \text{H}_2\text{O}$ .	$\text{C}_{12}\text{H}_{22}\text{O}_{11} + \text{H}_2\text{O}$ .	$\text{C}_{12}\text{H}_{22}\text{O}_{11}$ .	$\text{C}_{12}\text{H}_{22}\text{O}_{11} + \text{H}_2\text{O}$ .	
175	155.5	77.4	80.1	78.4	72.2	111.9	114.8	117.7	136.7	143.9	175
176	156.3	77.8	80.6	78.8	72.7	112.5	115.5	118.4	137.5	144.7	176
177	157.2	78.3	81.0	79.3	73.2	113.2	116.1	119.1	138.3	145.5	177
178	158.1	78.8	81.5	79.8	73.7	113.8	116.8	119.8	139.1	146.4	178
179	159.0	79.2	82.0	80.3	74.2	114.5	117.5	120.5	139.8	147.2	179
180	159.9	79.7	82.5	80.8	74.6	115.1	118.2	121.2	140.6	148.0	180
181	160.8	80.1	82.9	81.3	75.1	115.8	118.8	121.9	141.4	148.9	181
182	161.7	80.6	83.4	81.7	75.6	116.5	119.5	122.6	142.2	149.7	182
183	162.6	81.1	83.9	82.2	76.1	117.1	120.2	123.3	143.0	150.5	183
184	163.4	81.5	84.4	82.7	76.6	117.8	120.9	123.9	143.8	151.4	184
185	164.3	82.0	84.9	83.2	77.1	118.4	121.5	124.6	144.6	152.2	185
186	165.2	82.5	85.3	83.7	77.6	119.1	122.2	125.3	145.4	153.0	186
187	166.1	82.9	85.8	84.2	78.0	119.7	122.9	126.0	146.2	153.9	187
188	167.0	83.4	86.3	84.6	78.5	120.4	123.5	126.7	147.0	154.7	188
189	167.9	83.9	86.8	85.1	79.0	121.0	124.2	127.4	147.8	155.5	189
190	168.8	84.3	87.2	85.6	79.5	121.7	124.9	128.1	148.6	156.4	190
191	169.7	84.8	87.7	86.1	80.0	122.3	125.5	128.8	149.3	157.2	191
192	170.5	85.3	88.2	86.6	80.5	123.0	126.2	129.5	150.1	158.0	192
193	171.4	85.7	88.7	87.1	81.0	123.6	126.9	130.1	150.9	158.9	193
194	172.3	86.2	89.2	87.6	81.4	124.3	127.6	130.8	151.7	159.7	194
195	173.2	86.7	89.6	88.0	81.9	125.0	128.2	131.5	152.5	160.5	195
196	174.1	87.1	90.1	88.5	82.4	125.6	128.9	132.2	153.3	161.4	196
197	175.0	87.6	90.6	89.0	82.9	126.3	129.6	132.9	154.1	162.2	197
198	175.9	88.1	91.1	89.5	83.4	126.9	130.3	133.6	154.9	163.0	198
199	176.8	88.5	91.6	90.0	83.9	127.6	130.9	134.3	155.7	163.9	199
200	177.7	89.0	92.0	90.5	84.4	128.2	131.6	135.0	156.5	164.7	200
201	178.5	89.5	92.5	91.0	84.8	128.9	132.3	135.7	157.3	165.5	201
202	179.4	89.9	93.0	91.4	85.3	129.5	132.9	136.3	158.1	166.4	202
203	180.3	90.4	93.5	91.9	85.8	130.2	133.6	137.0	158.8	167.2	203
204	181.2	90.9	94.0	92.4	86.3	130.8	134.3	137.7	159.6	168.0	204
205	182.1	91.4	94.5	92.9	86.8	131.5	135.0	138.4	160.4	168.9	205
206	183.0	91.8	94.9	93.4	87.3	132.1	135.6	139.1	161.2	169.7	206
207	183.9	92.3	95.4	93.9	87.8	132.8	136.3	139.8	162.0	170.5	207
208	184.8	92.8	95.9	94.4	88.3	133.4	137.0	140.5	162.8	171.4	208
209	185.6	93.2	96.4	94.9	88.8	134.1	137.6	141.2	163.6	172.2	209
210	186.5	93.7	96.9	95.4	89.2	134.8	138.3	141.9	164.4	173.0	210
211	187.4	94.2	97.4	95.8	89.7	135.4	139.0	142.5	165.2	173.8	211
212	188.3	94.6	97.8	96.3	90.2	136.1	139.6	143.2	166.0	174.7	212
213	189.2	95.1	98.3	96.8	90.7	136.7	140.3	143.9	166.8	175.5	213
214	190.1	95.6	98.8	97.3	91.2	137.4	141.0	144.6	167.5	176.4	214
215	191.0	96.1	99.3	97.8	91.7	138.0	141.7	145.3	168.3	177.2	215
216	191.9	96.5	99.8	98.3	92.2	138.7	142.3	146.0	169.1	178.0	216
217	192.8	97.0	100.3	98.8	92.7	139.3	143.0	146.7	169.9	178.9	217
218	193.6	97.5	100.8	99.3	93.2	140.0	143.7	147.3	170.7	179.7	218
219	194.5	98.0	101.2	99.8	93.7	140.6	144.3	148.0	171.5	180.5	219
220	195.4	98.4	101.7	100.3	94.2	141.3	145.0	148.7	172.3	181.4	220
221	196.3	98.9	102.2	100.8	94.7	141.9	145.7	149.4	173.1	182.2	221
222	197.2	99.4	102.7	101.2	95.1	142.6	146.3	150.1	173.9	183.0	222
223	198.1	99.9	103.2	101.7	95.6	143.2	147.0	150.8	174.7	183.9	223
224	199.0	100.3	103.7	102.2	96.1	143.9	147.7	151.5	175.5	184.7	224
225	199.9	100.8	104.2	102.7	96.6	144.6	148.4	152.2	176.2	185.5	225
226	200.7	101.3	104.6	103.2	97.1	145.2	149.0	152.9	177.0	186.4	226
227	201.6	101.8	105.1	103.7	97.6	145.9	149.7	153.6	177.8	187.2	227
228	202.5	102.2	105.6	104.2	98.1	146.5	150.4	154.2	178.6	188.0	228
229	203.4	102.7	106.1	104.7	98.6	147.2	151.1	154.9	179.4	188.8	229

Table for calculating dextrose, invert sugar alone, invert sugar in the presence of sucrose (0.4 gram and 2 grams total sugar), lactose (three forms), and maltose (anhydrous and crystallized)—Continued.

[Expressed in milligrams.]

Cuprous oxid ( $\text{Cu}_2\text{O}$ ).	Copper (Cu).	Dextrose ( <i>d</i> -glucose).	Invert sugar.	Invert sugar and sucrose.		Lactose.			Maltose.		Cuprous oxid ( $\text{Cu}_2\text{O}$ ).
				0.4 gram total sugar.	2 grams total sugar.	$\text{C}_6\text{H}_{12}\text{O}_6$ .	$\text{C}_6\text{H}_{12}\text{O}_6 + \frac{1}{2} \text{H}_2\text{O}$ .	$\text{C}_6\text{H}_{12}\text{O}_6 + \text{H}_2\text{O}$ .	$\text{C}_6\text{H}_{12}\text{O}_6$ .	$\text{C}_6\text{H}_{12}\text{O}_6 + \text{H}_2\text{O}$ .	
230	204.3	103.2	106.6	105.2	99.1	147.8	151.7	155.6	180.2	189.7	230
231	205.2	103.7	107.1	105.7	99.6	148.5	152.4	156.3	181.0	190.5	231
232	206.1	104.1	107.6	106.2	100.1	149.1	153.1	157.0	181.8	191.3	232
233	207.0	104.6	108.1	106.7	100.6	149.8	153.7	157.7	182.6	192.2	233
234	207.9	105.1	108.6	107.2	101.1	150.5	154.4	158.4	183.4	193.0	234
235	208.7	105.6	109.1	107.7	101.6	151.1	155.1	159.1	184.2	193.8	235
236	209.6	106.0	109.5	108.2	102.1	151.8	155.8	159.7	184.9	194.7	236
237	210.5	106.5	110.0	108.7	102.6	152.4	156.4	160.4	185.7	195.5	237
238	211.4	107.0	110.5	109.2	103.1	153.1	157.1	161.1	186.5	196.3	238
239	212.3	107.5	111.0	109.6	103.5	153.7	157.8	161.8	187.3	197.2	239
240	213.2	108.0	111.5	110.1	104.0	154.4	158.4	162.5	188.1	198.0	240
241	214.1	108.4	112.0	110.6	104.5	155.0	159.1	163.2	188.9	198.8	241
242	215.0	108.9	112.5	111.1	105.0	155.7	159.8	163.9	189.7	199.7	242
243	215.8	109.4	113.0	111.6	105.5	156.3	160.5	164.6	190.5	200.5	243
244	216.7	109.9	113.5	112.1	106.0	157.0	161.1	165.3	191.3	201.3	244
245	217.6	110.4	114.0	112.6	106.5	157.7	161.8	166.0	192.1	202.2	245
246	218.5	110.8	114.5	113.1	107.0	158.3	162.5	166.6	192.9	203.0	246
247	219.4	111.3	115.0	113.6	107.5	159.0	163.1	167.3	193.6	203.8	247
248	220.3	111.8	115.4	114.1	108.0	159.6	163.8	168.0	194.4	204.7	248
249	221.2	112.3	115.9	114.6	108.5	160.3	164.5	168.7	195.2	205.5	249
250	222.1	112.8	116.4	115.1	109.0	160.9	165.2	169.4	196.0	206.3	250
251	223.0	113.2	116.9	115.6	109.5	161.6	165.8	170.1	196.8	207.2	251
252	223.8	113.7	117.4	116.1	110.0	162.2	166.5	170.8	197.6	208.0	252
253	224.7	114.2	117.9	116.6	110.5	162.9	167.2	171.5	198.4	208.8	253
254	225.6	114.7	118.4	117.1	111.0	163.5	167.9	172.2	199.2	209.7	254
255	226.5	115.2	118.9	117.6	111.5	164.2	168.5	172.8	200.0	210.5	255
256	227.4	115.7	119.4	118.1	112.0	164.8	169.2	173.5	200.8	211.3	256
257	228.3	116.1	119.9	118.6	112.5	165.5	169.9	174.2	201.6	212.2	257
258	229.2	116.6	120.4	119.1	113.0	166.2	170.5	174.9	202.3	213.0	258
259	230.1	117.1	120.9	119.6	113.5	166.8	171.2	175.6	203.1	213.8	259
260	231.0	117.6	121.4	120.1	114.0	167.5	171.9	176.3	203.9	214.7	260
261	231.8	118.1	121.9	120.6	114.5	168.1	172.5	177.0	204.7	215.5	261
262	232.7	118.6	122.4	121.1	115.0	168.8	173.2	177.7	205.5	216.3	262
263	233.6	119.0	122.9	121.6	115.5	169.4	173.9	178.3	206.3	217.2	263
264	234.5	119.5	123.4	122.1	116.0	170.1	174.6	179.0	207.1	218.0	264
265	235.4	120.0	123.9	122.6	116.5	170.7	175.2	179.7	207.9	218.8	265
266	236.3	120.5	124.4	123.1	117.0	171.4	175.9	180.4	208.7	219.7	266
267	237.2	121.0	124.9	123.6	117.5	172.0	176.6	181.1	209.5	220.5	267
268	238.1	121.5	125.4	124.1	118.0	172.7	177.2	181.8	210.3	221.3	268
269	238.9	122.0	125.9	124.6	118.5	173.3	177.9	182.5	211.0	222.1	269
270	239.8	122.5	126.4	125.1	119.0	174.0	178.6	183.2	211.8	223.0	270
271	240.7	122.9	126.9	125.6	119.5	174.6	179.2	183.8	212.6	223.8	271
272	241.6	123.4	127.4	126.2	120.0	175.3	179.9	184.5	213.4	224.6	272
273	242.5	123.9	127.9	126.7	120.6	176.0	180.6	185.2	214.2	225.5	273
274	243.4	124.4	128.4	127.2	121.1	176.6	181.3	185.9	215.0	226.3	274
275	244.3	124.9	128.9	127.7	121.6	177.3	181.9	186.6	215.8	227.1	275
276	245.2	125.4	129.4	128.2	122.1	177.9	182.6	187.3	216.6	228.0	276
277	246.1	125.9	129.9	128.7	122.6	178.6	183.3	188.0	217.4	228.8	277
278	246.9	126.4	130.4	129.2	123.1	179.2	184.0	188.7	218.2	229.6	278
279	247.8	126.9	130.9	129.7	123.6	179.9	184.6	189.4	218.9	230.5	279
280	248.7	127.3	131.4	130.2	124.1	180.6	185.3	190.1	219.7	231.3	280
281	249.6	127.8	131.9	130.7	124.6	181.2	186.0	190.7	220.5	232.1	281
282	250.5	128.3	132.4	131.2	125.1	181.9	186.6	191.4	221.3	233.0	282
283	251.4	128.8	132.9	131.7	125.6	182.5	187.3	192.1	222.1	233.8	283
284	252.3	129.3	133.4	132.2	126.1	183.2	188.0	192.8	222.9	234.6	284

Table for calculating dextrose, invert sugar alone, invert sugar in the presence of sucrose (0.4 gram and 2 grams total sugar), lactose (three forms), and maltose (anhydrous and crystallized)—Continued.

[Expressed in milligrams.]

Cuprous oxid ( $\text{Cu}_2\text{O}$ ).	Copper (Cu).	Dextrose ( <i>d</i> -glucose).	Invert sugar.	Invert sugar and sucrose.		Lactose.			Maltose.		Cuprous oxid ( $\text{Cu}_2\text{O}$ ).
				0.4 gram total sugar.	2 grams total sugar.	$\text{C}_{12}\text{H}_{22}\text{O}_{11}$ .	$\text{C}_{12}\text{H}_{22}\text{O}_{11} + \frac{1}{2} \text{H}_2\text{O}$ .	$\text{C}_{12}\text{H}_{22}\text{O}_{11} + \text{H}_2\text{O}$ .	$\text{C}_{12}\text{H}_{22}\text{O}_{11}$ .	$\text{C}_{12}\text{H}_{22}\text{O}_{11} + \text{H}_2\text{O}$ .	
285	253.2	129.8	133.9	132.7	126.6	183.8	188.7	193.5	223.7	235.5	285
286	254.0	130.3	134.4	133.2	127.1	184.5	189.3	194.2	224.5	236.3	286
287	254.9	130.8	134.9	133.7	127.6	185.1	190.0	194.9	225.3	237.1	287
288	255.8	131.3	135.4	134.3	128.1	185.8	190.7	195.5	226.1	238.0	288
289	256.7	131.8	135.9	134.8	128.6	186.4	191.3	196.2	226.9	238.8	289
290	257.6	132.3	136.4	135.3	129.2	187.1	192.0	196.9	227.6	239.6	290
291	258.5	132.7	136.9	135.8	129.7	187.7	192.7	197.6	228.4	240.5	291
292	259.4	133.2	137.4	136.3	130.2	188.4	193.3	198.3	229.2	241.3	292
293	260.3	133.7	137.9	136.8	130.7	189.0	194.0	199.0	230.0	242.1	293
294	261.2	134.2	138.4	137.3	131.2	189.7	194.7	199.7	230.8	242.9	294
295	262.0	134.7	138.9	137.8	131.7	190.3	195.4	200.4	231.6	243.8	295
296	262.9	135.2	139.4	138.3	132.2	191.0	196.0	201.0	232.4	244.6	296
297	263.8	135.7	140.0	138.8	132.7	191.7	196.7	201.7	233.2	245.4	297
298	264.7	136.2	140.5	139.4	133.2	192.3	197.4	202.4	234.0	246.3	298
299	265.6	136.7	141.0	139.9	133.7	193.0	198.0	203.1	234.8	247.1	299
300	266.5	137.2	141.5	140.4	134.2	193.6	198.7	203.8	235.5	247.9	300
301	267.4	137.7	142.0	140.9	134.8	194.3	199.4	204.5	236.3	248.8	301
302	268.3	138.2	142.5	141.4	135.3	194.9	200.0	205.2	237.1	249.6	302
303	269.1	138.7	143.0	141.9	135.8	195.6	200.7	205.9	237.9	250.4	303
304	270.0	139.2	143.5	142.4	136.3	196.2	201.4	206.5	238.7	251.3	304
305	270.9	139.7	144.0	142.9	136.8	196.9	202.1	207.2	239.5	252.1	305
306	271.8	140.2	144.5	143.4	137.3	197.5	202.7	207.9	240.3	252.9	306
307	272.7	140.7	145.0	144.0	137.8	198.2	203.4	208.6	241.1	253.8	307
308	273.6	141.2	145.5	144.5	138.3	198.8	204.1	209.3	241.9	254.6	308
309	274.5	141.7	146.1	145.0	138.8	199.5	204.7	210.0	242.7	255.4	309
310	275.4	142.2	146.6	145.5	139.4	200.1	205.4	210.7	243.5	256.3	310
311	276.3	142.7	147.1	146.0	139.9	200.8	206.1	211.4	244.2	257.1	311
312	277.1	143.2	147.6	146.5	140.4	201.4	206.7	212.1	245.0	257.9	312
313	278.0	143.7	148.1	147.0	140.9	202.1	207.4	212.7	245.8	258.8	313
314	278.9	144.2	148.6	147.6	141.4	202.8	208.1	213.4	246.6	259.6	314
315	279.8	144.7	149.1	148.1	141.9	203.4	208.8	214.1	247.4	260.4	315
316	280.7	145.2	149.6	148.6	142.4	204.1	209.4	214.8	248.2	261.2	316
317	281.6	145.7	150.1	149.1	143.0	204.7	210.1	215.5	249.0	262.1	317
318	282.5	146.2	150.7	149.6	143.5	205.4	210.8	216.2	249.8	262.9	318
319	283.4	146.7	151.2	150.1	144.0	206.0	211.5	216.9	250.6	263.7	319
320	284.2	147.2	151.7	150.7	144.5	206.7	212.1	217.6	251.3	264.6	320
321	285.1	147.7	152.2	151.2	145.0	207.3	212.8	218.3	252.1	265.4	321
322	286.0	148.2	152.7	151.7	145.5	208.0	213.5	218.9	252.9	266.2	322
323	286.9	148.7	153.2	152.2	146.0	208.6	214.1	219.6	253.7	267.1	323
324	287.8	149.2	153.7	152.7	146.6	209.3	214.8	220.3	254.5	267.9	324
325	288.7	149.7	154.3	153.2	147.1	210.0	215.5	221.0	255.3	268.7	325
326	289.6	150.2	154.8	153.8	147.6	210.6	216.2	221.7	256.1	269.6	326
327	290.5	150.7	155.3	154.3	148.1	211.3	216.8	222.4	256.9	270.4	327
328	291.4	151.2	155.8	154.8	148.6	211.9	217.5	223.1	257.7	271.2	328
329	292.2	151.7	156.3	155.3	149.1	212.6	218.2	223.8	258.5	272.1	329
330	293.1	152.2	156.8	155.8	149.7	213.2	218.8	224.4	259.3	272.9	330
331	294.0	152.7	157.3	156.4	150.2	213.9	219.5	225.1	260.0	273.7	331
332	294.9	153.2	157.9	156.9	150.7	214.5	220.2	225.8	260.8	274.6	332
333	295.8	153.7	158.4	157.4	151.2	215.2	220.8	226.5	261.6	275.4	333
334	296.7	154.2	158.9	157.9	151.7	215.8	221.5	227.2	262.4	276.2	334
335	297.6	154.7	159.4	158.4	152.3	216.5	222.2	227.9	263.2	277.0	335
336	298.5	155.2	159.9	159.0	152.8	217.1	222.9	228.6	264.0	277.9	336
337	299.3	155.8	160.5	159.5	153.3	217.8	223.5	229.2	264.8	278.7	337
338	300.2	156.3	161.0	160.0	153.8	218.4	224.2	229.9	265.6	279.5	338
339	301.1	156.8	161.5	160.5	154.3	219.1	224.9	230.6	266.4	280.4	339



Table for calculating dextrose, invert sugar alone, invert sugar in the presence of sucrose (0.4 gram and 2 grams total sugar), lactose (three forms), and maltose (anhydrous and crystallized)—Continued.

[Expressed in milligrams.]

Cuprous oxid ( $\text{Cu}_2\text{O}$ ).	Copper (Cu).	Dextrose ( <i>d</i> -glucose).	Invert sugar.	Invert sugar and sucrose.		Lactose.			Maltose.		Cuprous oxid ( $\text{Cu}_2\text{O}$ ).
				0.4 gram total sugar.	2 grams total sugar.	$\text{C}_{12}\text{H}_{22}\text{O}_{11}$ .	$\text{C}_{12}\text{H}_{22}\text{O}_{11} + \frac{1}{2}\text{H}_2\text{O}$ .	$\text{C}_{12}\text{H}_{22}\text{O}_{11} + \text{H}_2\text{O}$ .	$\text{C}_{12}\text{H}_{22}\text{O}_{11}$ .	$\text{C}_{12}\text{H}_{22}\text{O}_{11} + \text{H}_2\text{O}$ .	
340	302.0	157.3	162.0	161.0	154.8	219.8	225.5	231.3	267.1	281.2	340
341	302.9	157.8	162.5	161.6	155.4	220.4	226.2	232.0	267.9	282.0	341
342	303.8	158.3	163.1	162.1	155.9	221.1	226.9	232.7	268.7	282.9	342
343	304.7	158.8	163.6	162.6	156.4	221.7	227.5	233.4	269.5	283.7	343
344	305.6	159.3	164.1	163.1	156.9	222.4	228.2	234.1	270.3	284.5	344
345	306.5	159.8	164.6	163.7	157.5	223.0	228.9	234.7	271.1	285.4	345
346	307.3	160.3	165.1	164.2	158.0	223.7	229.6	235.4	271.9	286.2	346
347	308.2	160.8	165.7	164.7	158.5	224.3	230.2	236.1	272.7	287.0	347
348	309.1	161.4	166.2	165.2	159.0	225.0	230.9	236.8	273.5	287.9	348
349	310.0	161.9	166.7	165.7	159.5	225.6	231.6	237.5	274.3	288.7	349
350	310.9	162.4	167.2	166.3	160.1	226.3	232.2	238.2	275.0	289.5	350
351	311.8	162.9	167.7	166.8	160.6	226.9	232.9	238.9	275.8	290.4	351
352	312.7	163.4	168.3	167.3	161.1	227.6	233.6	239.6	276.6	291.2	352
353	313.6	163.9	168.8	167.8	161.6	228.2	234.2	240.2	277.4	292.0	353
354	314.4	164.4	169.3	168.4	162.2	228.9	234.9	240.9	278.2	292.8	354
355	315.3	164.9	169.8	168.9	162.7	229.5	235.6	241.6	279.0	293.7	355
356	316.2	165.4	170.4	169.4	163.2	230.2	236.3	242.3	279.8	294.5	356
357	317.1	166.0	170.9	170.0	163.7	230.8	236.9	243.0	280.6	295.3	357
358	318.0	166.5	171.4	170.5	164.3	231.5	237.6	243.7	281.4	296.2	358
359	318.9	167.0	171.9	171.0	164.8	232.1	238.3	244.4	282.2	297.0	359
360	319.8	167.5	172.5	171.5	165.3	232.8	238.9	245.1	282.9	297.8	360
361	320.7	168.0	173.0	172.1	165.8	233.5	239.6	245.8	283.7	298.7	361
362	321.6	168.5	173.5	172.6	166.4	234.1	240.3	246.4	284.5	299.5	362
363	322.4	169.0	174.0	173.1	166.9	234.8	241.0	247.1	285.3	300.3	363
364	323.3	169.6	174.6	173.7	167.4	235.4	241.6	247.8	286.1	301.2	364
365	324.2	170.1	175.1	174.2	167.9	236.1	242.3	248.5	286.9	302.0	365
366	325.1	170.6	175.6	174.7	168.5	236.7	243.0	249.2	287.7	302.8	366
367	326.0	171.1	176.1	175.2	169.0	237.4	243.6	249.9	288.5	303.6	367
368	326.9	171.6	176.7	175.8	169.5	238.1	244.3	250.6	289.3	304.5	368
369	327.8	172.1	177.2	176.3	170.0	238.7	245.0	251.3	290.0	305.3	369
370	328.7	172.7	177.7	176.8	170.6	239.4	245.7	252.0	290.8	306.1	370
371	329.5	173.2	178.3	177.4	171.1	240.0	246.3	252.7	291.6	307.0	371
372	330.4	173.7	178.8	177.9	171.6	240.7	247.0	253.3	292.4	307.8	372
373	331.3	174.2	179.3	178.4	172.2	241.3	247.7	254.0	293.2	308.6	373
374	332.2	174.7	179.8	179.0	172.7	242.0	248.4	254.7	294.0	309.5	374
375	333.1	175.3	180.4	179.5	173.2	242.6	249.0	255.4	294.8	310.3	375
376	334.0	175.8	180.9	180.0	173.7	243.3	249.7	256.1	295.6	311.1	376
377	334.9	176.3	181.4	180.6	174.3	243.9	250.4	256.8	296.4	312.0	377
378	335.8	176.8	182.0	181.1	174.8	244.6	251.0	257.5	297.2	312.8	378
379	336.7	177.3	182.5	181.6	175.3	245.2	251.7	258.2	297.9	313.6	379
380	337.5	177.9	183.0	182.1	175.9	245.9	252.4	258.8	298.7	314.5	380
381	338.4	178.4	183.6	182.7	176.4	246.6	253.0	259.5	299.5	315.3	381
382	339.3	178.9	184.1	183.2	176.9	247.2	253.7	260.2	300.3	316.1	382
383	340.2	179.4	184.6	183.8	177.5	247.9	254.4	260.9	301.1	316.9	383
384	341.1	180.0	185.2	184.3	178.0	248.5	255.1	261.6	301.9	317.8	384
385	342.0	180.5	185.7	184.8	178.5	249.2	255.7	262.3	302.7	318.6	385
386	342.9	181.0	186.2	185.4	179.1	249.8	256.4	263.0	303.5	319.4	386
387	343.8	181.5	186.8	185.9	179.6	250.5	257.1	263.6	304.2	320.3	387
388	344.6	182.0	187.3	186.4	180.1	251.1	257.7	264.3	305.0	321.1	388
389	345.5	182.6	187.8	187.0	180.6	251.8	258.4	265.0	305.8	321.9	389
390	346.4	183.1	188.4	187.5	181.2	252.4	259.1	265.7	306.6	322.8	390
391	347.3	183.6	188.9	188.0	181.7	253.1	259.7	266.4	307.4	323.6	391
392	348.2	184.1	189.4	188.6	182.3	253.7	260.4	267.1	308.2	324.4	392
393	349.1	184.7	190.0	189.1	182.8	254.4	261.1	267.8	309.0	325.2	393
394	350.0	185.2	190.5	189.7	183.3	255.0	261.8	268.5	309.8	326.1	394

Table for calculating dextrose, invert sugar alone, invert sugar in the presence of sucrose (0.4 gram and 2 grams total sugar), lactose (three forms), and maltose (anhydrous and crystallized)—Continued.

[Expressed in milligrams.]

Cuprous oxid ( $\text{Cu}_2\text{O}$ ).	Copper (Cu).	Dextrose ( $d$ -glucose.).	Invert sugar.	Invert sugar and sucrose.		Lactose.			Maltose.		Cuprous oxid ( $\text{Cu}_2\text{O}$ ).
				0.4 gram total sugar.	2 grams total sugar.	$\text{C}_{12}\text{H}_{22}\text{O}_{11}$ .	$\text{C}_{12}\text{H}_{22}\text{O}_{11} + \frac{1}{2}\text{H}_2\text{O}$ .	$\text{C}_{12}\text{H}_{22}\text{O}_{11} + \text{H}_2\text{O}$ .	$\text{C}_{12}\text{H}_{22}\text{O}_{11}$ .	$\text{C}_{12}\text{H}_{22}\text{O}_{11} + \text{H}_2\text{O}$ .	
395	350.9	185.7	191.0	190.2	183.9	255.7	262.4	269.1	310.6	326.9	395
396	351.8	186.2	191.6	190.7	184.4	256.3	263.1	269.8	311.4	327.7	396
397	352.6	186.8	192.1	191.3	184.9	257.0	263.8	270.5	312.1	328.6	397
398	353.5	187.3	192.7	191.8	185.5	257.7	264.4	271.2	312.9	329.4	398
399	354.4	187.8	193.2	192.3	186.0	258.3	265.1	271.9	313.7	330.2	399
400	355.3	188.4	193.7	192.9	186.5	259.0	265.8	272.6	314.5	331.1	400
401	356.2	188.9	194.3	193.4	187.1	259.6	266.4	273.3	315.3	331.9	401
402	357.1	189.4	194.8	194.0	187.6	260.3	267.1	274.0	316.1	332.7	402
403	358.0	189.9	195.4	194.5	188.1	260.9	267.8	274.6	316.9	333.6	403
404	358.9	190.5	195.9	195.0	188.7	261.6	268.5	275.3	317.7	334.4	404
405	359.7	191.0	196.4	195.6	189.2	262.2	269.1	276.0	318.5	335.2	405
406	360.6	191.5	197.0	196.1	189.8	262.9	269.8	276.7	319.2	336.0	406
407	361.5	192.1	197.5	196.7	190.3	263.5	270.5	277.4	320.0	336.9	407
408	362.4	192.6	198.1	197.2	190.8	264.2	271.1	278.1	320.8	337.7	408
409	363.3	193.1	198.6	197.7	191.4	264.8	271.8	278.8	321.6	338.5	409
410	364.2	193.7	199.1	198.3	191.9	265.5	272.5	279.5	322.4	339.4	410
411	365.1	194.2	199.7	198.8	192.5	266.1	273.1	280.1	323.2	340.2	411
412	366.0	194.7	200.2	199.4	193.0	266.8	273.8	280.8	324.0	341.0	412
413	366.9	195.2	200.8	199.9	193.5	267.4	274.5	281.5	324.8	341.9	413
414	367.7	195.8	201.3	200.5	194.1	268.1	275.2	282.2	325.6	342.7	414
415	368.6	196.3	201.8	201.0	194.6	268.7	275.8	282.9	326.3	343.5	415
416	369.5	196.8	202.4	201.6	195.2	269.4	276.5	283.6	327.1	344.4	416
417	370.4	197.4	202.9	202.1	195.7	270.1	277.2	284.3	327.9	345.2	417
418	371.3	197.9	203.5	202.6	196.2	270.7	277.8	285.0	328.7	346.0	418
419	372.2	198.4	204.0	203.2	196.8	271.4	278.5	285.6	329.5	346.8	419
420	373.1	199.0	204.6	203.7	197.3	272.0	279.2	286.3	330.3	347.7	420
421	374.0	199.5	205.1	204.3	197.9	272.7	279.8	287.0	331.1	348.5	421
422	374.8	200.1	205.7	204.8	198.4	273.3	280.5	287.7	331.9	349.3	422
423	375.7	200.6	206.2	205.4	198.9	274.0	281.2	288.4	332.7	350.2	423
424	376.6	201.1	206.7	205.9	199.5	274.6	281.9	289.1	333.4	351.0	424
425	377.5	201.7	207.3	206.5	200.0	275.3	282.5	289.8	334.2	351.8	425
426	378.4	202.2	207.8	207.0	200.6	275.9	283.2	290.5	335.0	352.7	426
427	379.3	202.8	208.4	207.6	201.1	276.6	283.9	291.1	335.8	353.5	427
428	380.2	203.3	208.9	208.1	201.7	277.2	284.5	291.8	336.6	354.3	428
429	381.1	203.8	209.5	208.7	202.2	277.9	285.2	292.5	337.4	355.1	429
430	382.0	204.4	210.0	209.2	202.7	278.5	285.9	293.2	338.2	356.0	430
431	382.8	204.9	210.6	209.8	203.3	279.2	286.5	293.9	339.0	356.8	431
432	383.7	205.5	211.1	210.3	203.8	279.8	287.2	294.6	339.7	357.6	432
433	384.6	206.0	211.7	210.9	204.4	280.5	287.9	295.3	340.5	358.5	433
434	385.5	206.5	212.2	211.4	204.9	281.2	288.6	295.9	341.3	359.3	434
435	386.4	207.1	212.8	212.0	205.5	281.8	289.2	296.6	342.1	360.1	435
436	387.3	207.6	213.3	212.5	206.0	282.5	289.9	297.3	342.9	361.0	436
437	388.2	208.2	213.9	213.1	206.6	283.1	290.6	298.0	343.7	361.8	437
438	389.1	208.7	214.4	213.6	207.1	283.8	291.2	298.7	344.5	362.6	438
439	390.0	209.2	215.0	214.2	207.7	284.4	291.9	299.4	345.3	363.4	439
440	390.8	209.8	215.5	214.7	208.2	285.1	292.6	300.1	346.1	364.3	440
441	391.7	210.3	216.1	215.3	208.8	285.7	293.2	300.8	346.8	365.1	441
442	392.6	210.9	216.6	215.8	209.3	286.4	293.9	301.4	347.6	365.9	442
443	393.5	211.4	217.2	216.4	209.9	287.0	294.6	302.1	348.4	366.8	443
444	394.4	212.0	217.8	216.9	210.4	287.7	295.3	302.8	349.2	367.6	444
445	395.3	212.5	218.3	217.5	211.0	288.3	295.9	303.5	350.0	368.4	445
446	396.2	213.1	218.9	218.0	211.5	289.0	296.6	304.2	350.8	369.3	446
447	397.1	213.6	219.4	218.6	212.1	289.6	297.3	304.9	351.6	370.1	447
448	397.9	214.1	220.0	219.1	212.6	290.3	297.9	305.6	352.4	370.9	448
449	398.8	214.7	220.5	219.7	213.2	290.9	298.6	306.3	353.2	371.7	449

Table for calculating dextrose, invert sugar alone, invert sugar in the presence of sucrose (0.4 gram and 2 grams total sugar), lactose (three forms), and maltose (anhydrous and crystallized)—Continued.

[Expressed in milligrams.]

Cuprous oxid ( $\text{Cu}_2\text{O}$ ).	Copper (Cu).	Dextrose (d-glucose).	Invert sugar.	Invert sugar and sucrose.		Lactose.			Maltose.		Cuprous oxid ( $\text{Cu}_2\text{O}$ ).
				0.4 gram total sugar.	2 grams total sugar.	$\text{C}_{12}\text{H}_{22}\text{O}_{11}$ .	$\text{C}_{12}\text{H}_{22}\text{O}_{11} + \frac{1}{2} \text{H}_2\text{O}$ .	$\text{C}_{12}\text{H}_{22}\text{O}_{11} + \text{H}_2\text{O}$ .	$\text{C}_{12}\text{H}_{22}\text{O}_{11}$ .	$\text{C}_{12}\text{H}_{22}\text{O}_{11} + \text{H}_2\text{O}$ .	
450	399.7	215.2	221.1	220.2	213.7	291.6	299.3	306.9	353.9	372.6	450
451	400.6	215.8	221.6	220.8	214.3	292.3	299.9	307.6	354.7	373.4	451
452	401.5	216.3	222.2	221.4	214.8	292.9	300.6	308.3	355.5	374.2	452
453	402.4	216.9	222.8	221.9	215.4	293.6	301.3	309.0	356.3	375.1	453
454	403.3	217.4	223.3	222.5	215.9	294.2	302.0	309.7	357.1	375.9	454
455	404.2	218.0	223.9	223.0	216.5	294.9	302.6	310.4	357.9	376.7	455
456	405.1	218.5	224.4	223.6	217.0	295.5	303.3	311.1	358.7	377.4	456
457	405.9	219.1	225.0	224.1	217.6	296.2	304.0	311.8	359.5	378.4	457
458	406.8	219.6	225.5	224.7	218.1	296.8	304.6	312.4	360.3	379.2	458
459	407.7	220.2	226.1	225.3	218.7	297.5	305.3	313.1	361.0	380.0	459
460	408.6	220.7	226.7	225.8	219.2	298.1	306.0	313.8	361.8	380.9	460
461	409.5	221.3	227.2	226.4	219.8	298.8	306.6	314.5	362.6	381.7	461
462	410.4	221.8	227.8	226.9	220.3	299.4	307.3	315.2	363.4	382.5	462
463	411.3	222.4	228.3	227.5	220.9	300.1	308.0	315.9	364.2	383.4	463
464	412.2	222.9	228.9	228.1	221.4	300.7	308.7	316.6	365.0	384.2	464
465	413.0	223.5	229.5	228.6	222.0	301.4	309.3	317.3	365.8	385.0	465
466	413.9	224.0	230.0	229.2	222.5	302.0	310.0	317.9	366.6	385.9	466
467	414.8	224.6	230.6	229.7	223.1	302.7	310.7	318.6	367.3	386.7	467
468	415.7	225.1	231.2	230.3	223.7	303.3	311.3	319.3	368.1	387.5	468
469	416.6	225.7	231.7	230.9	224.2	304.0	312.0	320.0	368.9	388.3	469
470	417.5	226.2	232.3	231.4	224.8	304.7	312.7	320.7	369.7	389.2	470
471	418.4	226.8	232.8	232.0	225.3	305.3	313.3	321.4	370.5	390.0	471
472	419.3	227.4	233.4	232.5	225.9	306.0	314.0	322.1	371.3	390.8	472
473	420.2	227.9	234.0	233.1	226.4	306.6	314.7	322.8	372.1	391.7	473
474	421.0	228.5	234.5	233.7	227.0	307.3	315.4	323.4	372.9	392.5	474
475	421.9	229.0	235.1	234.2	227.6	307.9	316.0	324.1	373.7	393.3	475
476	422.8	229.6	235.7	234.8	228.1	308.6	316.7	324.8	374.4	394.2	476
477	423.7	230.1	236.2	235.4	228.7	309.2	317.4	325.5	375.2	395.0	477
478	424.6	230.7	236.8	235.9	229.2	309.9	318.0	326.2	376.0	395.8	478
479	425.5	231.3	237.4	236.5	229.8	310.5	318.7	326.9	376.8	396.6	479
480	426.4	231.8	237.9	237.1	230.3	311.2	319.4	327.6	377.6	397.5	480
481	427.3	232.4	238.5	237.6	230.9	311.8	320.0	328.2	378.4	398.3	481
482	428.1	232.9	239.1	238.2	231.5	312.5	320.7	328.9	379.2	399.1	482
483	429.0	233.5	239.6	238.8	232.0	313.1	321.4	329.6	380.0	400.0	483
484	429.9	234.1	240.2	239.3	232.6	313.8	322.1	330.3	380.7	400.8	484
485	430.8	234.6	240.8	239.9	233.2	314.4	322.7	331.0	381.5	401.6	485
486	431.7	235.2	241.4	240.5	233.7	315.1	323.4	331.7	382.3	402.4	486
487	432.6	235.7	241.9	241.0	234.3	315.8	324.1	332.4	383.1	403.3	487
488	433.5	236.3	242.5	241.6	234.8	316.4	324.7	333.1	383.9	404.1	488
489	434.4	236.9	243.1	242.2	235.4	317.1	325.4	333.7	384.7	404.9	489
490	435.3	237.4	243.6	242.7	236.0	317.7	326.1	334.4	385.5	405.8	490

## **XVII. METHODS FOR THE ANALYSIS OF MEAT AND MEAT PRODUCTS.**

**Acidity.—Provisional.**

[Page 106.]

Titrate a solution or an aqueous extract of the meat, or meat preparation, with N/10 alkali, using phenolphthalein as indicator; or if the color prevents the use of this indicator, use litmus paper, with the understanding that the results are lower than when phenolphthalein is employed.

## **XVIII. METHODS FOR THE ANALYSIS OF DAIRY PRODUCTS.**

### **CONDENSED MILK.—PROVISIONAL.**

#### **Fat or Ether Extract—Double Extraction Method.**

[Page 123.]

Extract the solid residue of about 5 grams of a 40 per cent solution with ether in the usual manner; dry, leave tubes in a dish containing 500 cc of water or more, for two or three hours; dry, extract again for about five hours, and determine fat as directed under milk (p. 119 et seq.).

## XX. METHODS FOR THE ANALYSIS OF COCOA AND COCOA PRODUCTS.—PROVISIONAL.

[Page 148.]

### 1. Moisture.

Proceed as directed under "VI. General Methods," page 38.

### 2. Ash.

Proceed as directed under "VI. General Methods," page 38.

### 3. Soluble Ash.

Proceed as directed under "X. Saccharine Products," 3 (c), page 68.

### 4. Ash Insoluble in Acid.

Proceed as directed under "XXIV. Spices," section 5, page 162.

### 5. Alkalinity of Ash.

Treat the entire ash from 2 grams of the material as directed under "X. Saccharine Products," 3 (e), page 69.

### 6. Total Nitrogen.

Determine total nitrogen by the Kjeldahl method, "I. Fertilizers," 4, page 5.

### 7. Theobromin and Caffein.<sup>a</sup>

Boil 10 grams of the powdered material and 5 grams of calcined magnesia for 30 minutes with 300 cc of water. Filter by suction on a Buchner funnel, using a round disk of filter paper. Transfer the material and paper to the same flask used for the first boiling, add 150 cc of water and boil for 15 minutes. Filter as before and repeat the operation of boiling and filtering. Wash once or twice with hot water. Evaporate the united filtrates (with ignited quartz sand if sugar be present) to complete dryness in a "Hoffmeister Schälchen" or other suitable thin glass dish of about 300 cc capacity. Grind the dish with contents to a coarse powder in a mortar provided with a suitable cover to prevent mechanical loss. Transfer to the inner tube of a Tollens, Johnson, or Wiley fat extractor, and dry thoroughly in a water oven. Extract with chloroform for eight hours, or until the theobromin and caffein are completely removed, into a weighed flask.<sup>b</sup> Distil off the chloroform and dry the residue at 100° C. to con-

<sup>a</sup>Decker-Kunze method. *Schweiz. Wochens. Chem. Pharm.*, 1902, **40**: 527, 530, 541, 545, 553-557; abstract *Chem. Centrbl.*, 1903, **74**: 62.

<sup>b</sup>It is important that the material be thoroughly dry; that an extractor be used which permits of a hot extraction, and that a considerable volume of chloroform pass through the material.

stant weight. Treat the residue in the flask for some hours at room temperature with 50 cc of benzol. Filter through a small paper into a tared dish, evaporate to dryness and dry to constant weight at 100° C., thus obtaining the amount of caffein.

Determine the theobromin by Kunze's<sup>a</sup> method as follows:

Add to the filter paper 150 cc of water, enough ammonia water to make the solution slightly alkaline, and an excess of decinormal silver nitrate solution. Boil to half the original volume, add 75 cc of water and repeat the boiling. If the solution still contains the slightest trace of free ammonia repeat the process of adding water and boiling until the solution is perfectly neutral. Filter off the insoluble silver theobromin compound and wash with hot water. In the filtrate determine the excess of silver nitrate<sup>b</sup> by Volhard's method as directed under "III. Inorganic Plant Constituents," 6, page 23.

### 8. Other Nitrogenous Substances.

Add the percentages of nitrogen present as theobromin and as caffein,<sup>c</sup> subtract the sum from the percentage of total nitrogen and multiply the remainder by 6.25.

### 9. Crude Fiber.<sup>d</sup>

Proceed as directed under "VI. General Methods," 11, page 56, except that the fiber is filtered and weighed on a paper.

### 10. Crude Starch.<sup>e</sup>

Weigh 4 grams of the material if unsweetened, or 10 grams if sweetened, into a small wedgewood mortar, add 25 cc of ether and grind with a pestle. After the coarser material has settled decant off the ether together with the fine suspended matter on a 11 cm blue ribbon S. and S. paper. Repeat this treatment until no more coarse material remains. After the ether has evaporated from the filter, transfer the fat-free residue to the mortar by means of a jet of cold water and rub to an even paste, filtering on the paper previously employed. Repeat this process until all sugar is removed. In the case of sweetened products the filtrate should measure at least 500 cc. Conduct the hydrolysis of the residue as directed for "Starch" under "VI. General Methods," 8 (a), page 53, except that after neutralizing with sodium hydroxid, add 5 cc of basic lead acetate solution<sup>f</sup> before completing the volume to 250 cc. To 100 cc of the filtrate add 1 cc of 60 per cent sulphuric acid, filter off the lead sulphate and determine reducing matters in 25 cc of the filtrate as directed under "VI. General Methods," for "Reducing Sugars," 7 (b), (2), page 49. Determine copper by the direct weighing of the cuprous oxid, 7 (c), (6), page 53.

### 11. Pure Starch.

Remove fat and sugar from 4 grams of the material if unsweetened, or 10 grams if sweetened, as directed under "Crude Starch." Carefully wash the wet residue into a beaker with 100 cc of water, heat over asbestos to boiling with constant stirring, and

<sup>a</sup> Zts. anal. Chem., 1894, 33: 1.

<sup>b</sup> 1 cc of decinormal silver nitrate solution is equivalent to 0.01802 gram of theobromin.

<sup>c</sup> The percentage of theobromin multiplied by 0.311 and that of caffein multiplied by 0.289 give the respective percentages of nitrogen.

<sup>d</sup> In the analyses of commercial cocoa and other finely ground, pulverized cocoa products, the residue after fat extraction may be used directly for fiber determination. If, however, the material is at all granular, it must be reduced to an impalpable powder, otherwise the results will be much too high. The pulverization may be satisfactorily performed by grinding with ether as hereinafter described under "10. Crude Starch," removing the extracted residue with the hot 1.25 per cent sulphuric-acid solution.

<sup>e</sup> Copper-reducing matters by direct acid hydrolysis.

<sup>f</sup> Prepared as directed under "VI. General Methods," 6 (b), (1), page 40.

continue the boiling and stirring for thirty minutes. Replace the water lost by evaporation and immerse the beaker in a water bath kept at 55° to 60° C. When the liquid has cooled to the temperature of the bath, add 20 cc of a freshly prepared extract of malt <sup>a</sup> and digest the mixture for two hours with occasional stirring. Boil a second time for thirty minutes, dilute, cool, and digest as before with another 20 cc portion of malt extract. Heat again to boiling, cool, and transfer to a 250-cc flask. Add 3 cc of alumina cream, make up to the mark, and filter through a dry paper.<sup>b</sup> Conduct the hydrolysis of 200 cc of the filtrate and determine the reducing power of the resultant solution as directed under "Crude Starch" in the preceding section (10), except that the treatment with basic lead acetate solution and its removal with sulphuric acid are omitted. Introduce a correction for dextrose, due to the added malt extract, as determined by accompanying blank analyses.

### 12. Fat.

Desiccate 2 grams of the material for two or three days or until the moisture is practically removed. Extract with anhydrous ether in a Tollens, Johnson, or Wiley fat extractor until no more fat is removed. Grind and repeat the extraction. Allow the ether to evaporate and dry the residue at 100° C. until the weight is constant.

### 13. Sucrose and Lactose (Dubois's Method).<sup>c</sup>

Extract the fat from 13 grams of the sample by shaking and centrifuging twice with 100 cc of gasoline, separating the solvent by decantation. To the residue add 100 cc of water and shake for ten minutes. Add 5 cc of basic lead acetate solution (p. 40), filter, and remove the excess of lead. Allow 25 cc of this solution to stand over night to destroy birotation, and polarize. Multiply readings by 2. Invert 50 cc of the above filtrate as described on page 40 (c), nearly neutralizing the acid after cooling with sodium hydroxid solution, and make up to 100 cc. Bring to temperature at which direct readings were made and polarize, and also polarize at 86° in a water jacketed tube; multiply all invert readings by 4.

Calculate the approximate weight of sucrose and lactose present in the 13 grams by the following formulæ:

$$\text{Grams of sucrose} = \frac{(a-b) \frac{1.05}{t} \times 13}{142.66 - \frac{t}{2}}$$

$$\text{Grams of lactose} = \frac{c \times 1.264 \times 1.11 \times 1.05 \times 13}{100} = \frac{19.152c}{100}$$

a=direct readings, normal weight.

b=invert readings, normal weight.

c=invert readings, normal weight at 86° C.

From the total amount of sugar found by the above, obtain the value of x from the table below and calculate sugars as follows:

$$\frac{(a-b) \frac{1.05x}{t}}{142.66 - \frac{t}{2}} = \text{per cent of sucrose. } (1.473c)x = \text{per cent of lactose.}$$

2 grams of sugar in sample, x=101.2

4 grams of sugar in sample, x=102.5

6 grams of sugar in sample, x=103.6

8 grams of sugar in sample, x=104.8

10 grams of sugar in sample, x=106.05

15 grams of sugar in sample, x=109.40

20 grams of sugar in sample, x=112.40

<sup>a</sup> Prepared as directed under "VI. General Methods," 8 (b), (1), page 53.

<sup>b</sup> The residue on the paper at this point should show no signs of starch when examined microscopically.

<sup>c</sup> J. Amer. Chem. Soc., 1907, 29: 556.



**14. Other Nitrogen-free Substances.**

Subtract from 100 the sum of the percentages of moisture, ash, theobromin, caffeine, other nitrogenous substances, pure starch, and fat.

**15. Constants of the Fat.**

On the ether extract determine melting point as directed under "XIX. Edible Fats and Oils," 4 (a), page 133; index of refraction as directed under 3 (b) or 3 (c), pages 132 to 133, and iodine absorption by the Hübl process, as directed under 6, page 136.

## XXIX. METHODS FOR THE ANALYSIS OF DRUGS.

[Page 201.]

### 2. Determination of Alkaloids.

#### (a) TOTAL EXTRACTION METHOD.

Into a 200 cc flask weigh 10 grams of the powdered drug, add about 75 cc of ether-chloroform mixture (5 to 1 by volume), rotate, and add 5 cc of a mixture of 40 cc of strong ammonia water with 60 cc of alcohol, cork, shake well and often during one hour, and let stand over night. Transfer as much of the mixture as possible to a small percolator, the neck of which is provided with a purified cotton plug, receiving the percolate in a beaker or flask. Rinse the residue in the flask into the percolator with additional portions of the ether-chloroform mixture, packing it down moderately with a glass rod, and continue the percolation with the same mixture until the percolate does not give an alkaloidal reaction, or at most only an opalescence when 2 cc are evaporated, and the residue is treated with N/10 hydrochloric acid, the mixture filtered and the filtrate tested with Mayer's solution. Evaporate or distil the percolate to about 10 cc at a temperature not exceeding 70° C. Transfer to a separator, and rinse the vessel with two portions of 20 cc each of ether, alternating with two portions of 10 cc each of 2 per cent sulphuric acid. Shake out, making sure that the reaction is acid, and repeat the operation three times with 15 cc of the same strength of acid, filtering the acid solutions into another separator through a 7 cm filter, and rinsing the latter with a few cubic centimeters of water. Wash the acid solution with 10 cc of ether-chloroform mixture (1 to 3 by volume), discarding the latter, and if colored repeat until no more color is required. Make the solution alkaline with ammonia water and shake out with four successive portions of about 20 cc each of ether-chloroform mixture (1 to 3); collect the latter in a separator, wash with 5 cc of water, transfer the ether-chloroform mixture to a tared flask or beaker, rinsing the separator with a small portion of ether-chloroform, and evaporate the solvent at a temperature not exceeding 70° C.

(1) *Gravimetric.* Add 3 cc of ether, evaporate and dry to constant weight at a temperature not exceeding 70° C. Report weight of alkaloidal residue.

(2) *Volumetric.* Dissolve the residue in about 10 cc of neutral alcohol, add 25 cc of water, 3 to 15 drops of cochineal indicator solution, measure in from a burette a slight excess of N/50 sulphuric acid, mix intimately, and titrate back with N/50 potassium hydroxid. Report net amount of N/50 acid required.

#### (b) ALIQUOT METHOD.

Into a 200 cc flask weigh 15 grams of the powdered drug, add 150 cc of ether-chloroform mixture (5 to 1 by volume), cork, and shake often for several minutes. Add 10 cc of ammonia water (10 per cent), shake frequently during one hour, and let stand over night. Add 15 cc of water, or sufficient to agglomerate the drug, shake, let settle a few minutes, and then decant 75 cc of the clear solution into a graduated cylinder. Transfer the solution to a separator, rinsing the cylinder with a few cubic centimeters of ether-chloroform, and shake out with 20 cc (or sufficient to render acid) of 2 per cent

sulphuric acid, repeating the operation three times with 15 cc of the same strength acid, and collect the acid solutions in another separator. Wash the acid solution with 10 cc of ether-chloroform mixture (1 to 3 by volume), discarding the latter, and, if colored, repeat until no more color is acquired. Make the solution alkaline with ammonia water, and shake out with four successive portions of about 20 cc each of ether-chloroform mixture (1 to 3); collect the latter in a separator, wash with 5 cc of water, transfer the ether-chloroform mixture to a tared flask or beaker, rinsing the separator with a small portion of ether-chloroform, and evaporate the solvent at a temperature not exceeding 70° C.

(a) *Gravimetric.* Add 3 cc of ether, evaporate and dry to constant weight at a temperature not exceeding 70° C. Report weight of alkaloidal residue.

(b) *Volumetric.* Dissolve the residue in about 10 cc of neutral alcohol, add 25 cc of water, 5 to 15 drops of cochineal indicator solution, measure in from a burette a slight excess of N/50 sulphuric acid, mix intimately, and titrate back with N/50 potassium hydroxid. Report net amount of N/50 acid required.

Whenever iodeosin indicator is used, follow the directions of the U. S. Pharmacopœia VIII, page 542. Solutions standardized by means of cochineal should not be used with iodeosin without running a blank for comparison, as the indicators do not give the same neutral point.

[These methods and those referred to in the U. S. Pharmacopœia are applied to the analysis of the following drugs with the modifications noted]:

#### ACONITE ROOT.

*Method I.*—U. S. Pharmacopœia VIII, p. 28. Report the amount of N/10 acid required.

*Method II.*—Aliquot gravimetric. Use *ether* instead of ether-chloroform mixture in the final shaking out.

#### BELLADONNA LEAVES.

*Method I.*—U. S. Pharmacopœia VIII, p. 67. Report indicator used and amount of N/10 acid required.

*Method II.*—Aliquot volumetric.

#### BELLADONNA ROOT.

*Method I.*—U. S. Pharmacopœia VIII, p. 68. Report indicator used and amount of N/10 acid required.

*Method II.*—Aliquot volumetric.

#### CINCHONA BARK.

*Method I.*—U. S. Pharmacopœia VIII, p. 102. Report total and ether-soluble alkaloids.

*Method II.*—Total extraction, gravimetric.

#### COCA LEAVES.

*Method I.*—U. S. Pharmacopœia VIII, p. 106. Report indicator used and amount of N/10 acid required.

*Method II.*—Aliquot gravimetric. Use *ether* instead of ether-chloroform mixture throughout.

#### COLCHICUM CORM.

*Method I.*—U. S. Pharmacopœia VIII, p. 111. Report weight of alkaloidal residue.

*Method II.*—Exhaust 10 grams of the powdered drug by percolation with alcohol, add 25 cc of water to the solution and evaporate. Digest and stir the residue for several

minutes with 25 cc of petroleum ether and transfer the liquid to a beaker, rinsing the residue with a little fresh petroleum ether. Add to the beaker 20 cc of water and evaporate the petroleum ether. Gently warm the first residue with 10 cc of water, cool, and filter the solution through a small filter into a separator, rinsing the vessel and filter with several small portions of water, and finally with the water in the beaker. Shake out the liquid with four portions of 10 cc each of chloroform and evaporate the latter in a tared dish. Add 3 cc of alcohol and evaporate, repeat the operation and dry the residue to constant weight at a temperature not exceeding 70° C. Report weight of alkaloidal residue.

COLCHICUM SEED.

*Method I.*—U. S. Pharmacopœia VIII, p. 112. Report weight of alkaloidal residue.

*Method II.*—See Colchicum Corm. Method II.

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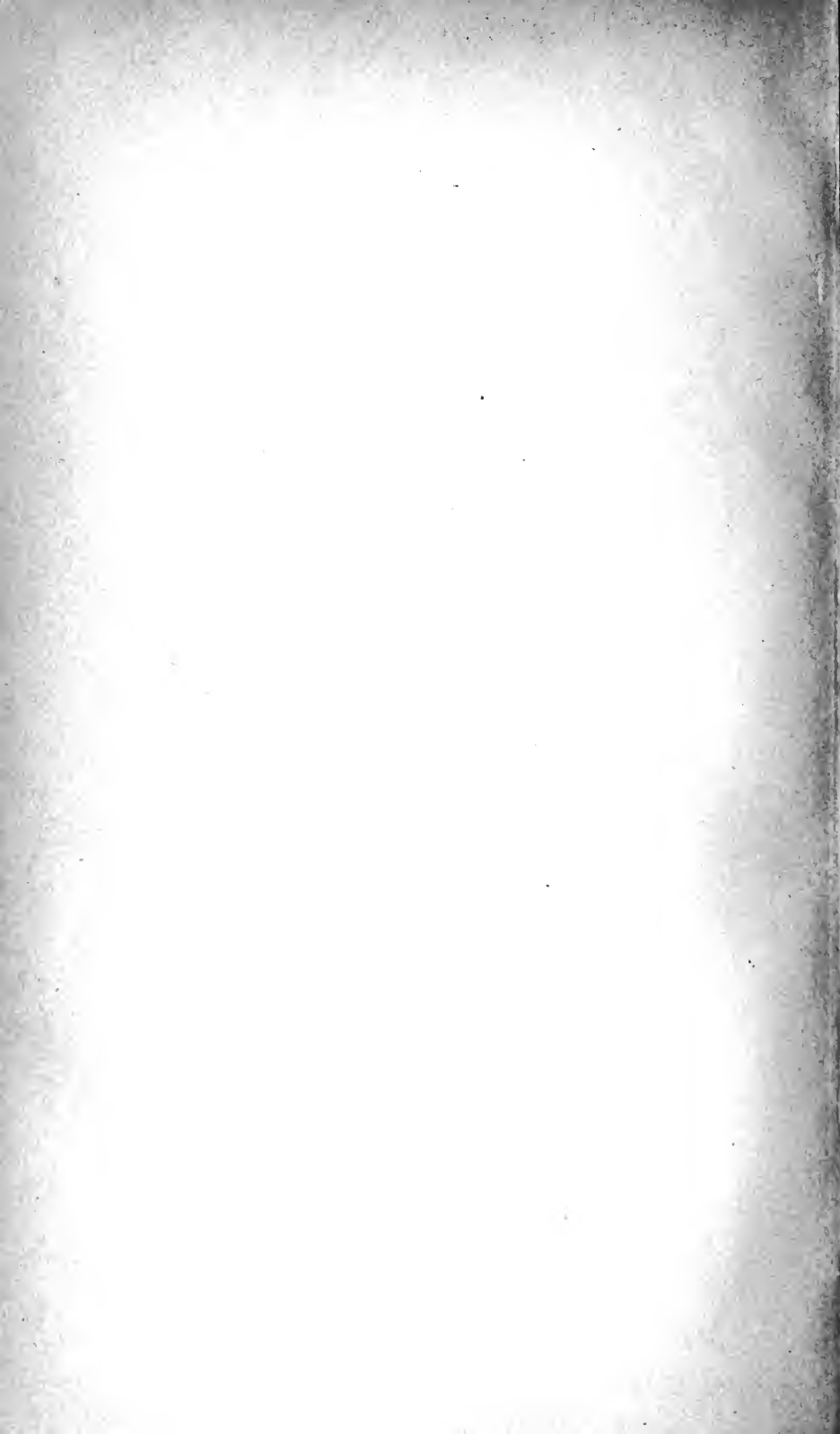
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